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Rayon Reports

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NEW METHOD FOR MEASURING YARN TENSION

Self-contained unit charts variables that affect tension

An improved method of recording yarn tensions has been announced by Avisco's Textile Research Department. The method utilizes a recording strain gauge developed from a basic idea submitted by Saco-Lowell Shops and has been specifically designed by Textile Research Department engineers for both laboratory research and general mill use.

The assembled equipment can be used on nearly any textile machine. It provides a reliable means of charting the results of any variable that affects tension—such as moisture, relative humidity, lubricants, guides and tensions or cyclic variations of twist or strain.

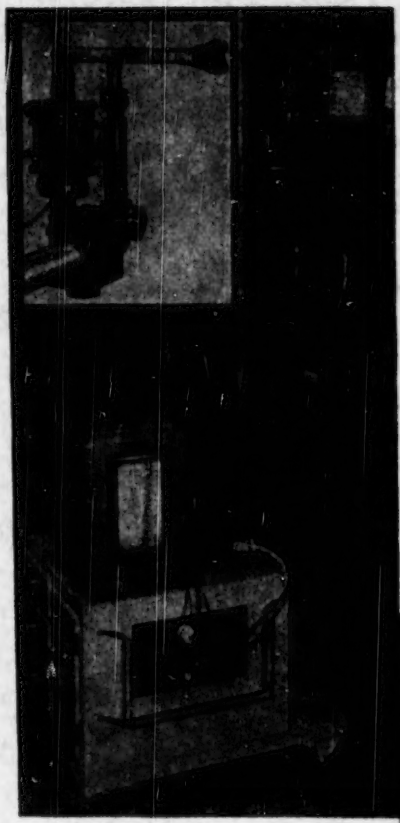
Strain gauge feeds electronic recorder

The tensiometer utilizes means of measurement well established in other mechanical fields. A strain gauge of great sensitivity, but with practically no inertia effect, feeds an electronic recorder.

Seen as adjunct to hand tensiometer

This electronic equipment is not intended to supplant the hand-held tensiometer in every day use. Its greatest use will be to study operations so that safe levels and uniformity of tension can be established. Proper control can then be maintained by use of the smaller devices. Results to date indicate its value in spot checks of various tension devices, finishes and a few operating conditions such as twisting, cake and cone winding, spooling, warping, quilling and tricot knitting. The equipment can also be used for yarn tension measurement on slashers, looms and adapted for use on spinning frames and for some fabric processing machinery.

The recording tensiometer was developed as part of Avisco's continuous program of service to the textile industry. Demonstrations will be arranged for interested parties at the Textile Research Department, Marcus Hook, Pa.



The new tensiometer in operation. Inset photo shows close-up of gauge itself.

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Textile Testing

The opening lecture was given by Mr. H. M. Taylor, M.A. (Cantab.), A.Inst.P. (British Cotton Industry Research Association) and entitled "Textile Testing." For the proper understanding of textile testing and also of the principles governing the drawing up of specifications for the final product, some understanding of statistical method is essential, said Mr. Taylor.

Among the aims of textile testing are: (1) The routine examination of raw material and the control of the processes of spinning, manufacture and finishing. (2) The inspection of goods on receipt to ensure that an agreed standard is being maintained and to reduce the possibility of substandard goods being put into service. (3) The investigation of faults in textile products to determine the origin of defects. (4) The assessment of utility and serviceability in new products.

Under (2) the principles that underlie acceptance and the drawing up of specifications were outlined and tests that characterise features of general and special importance in yarns and fabrics were discussed. Under (3) methods that can be applied to the examination of faults of the more common types in yarns and fabrics were described. Mr. Taylor concluded by saying that the assessment of utility and serviceability in textiles is one of the most difficult of the tasks of the textile technologist. Progress is being made slowly, mainly on a few specialised fronts.

FROM THE BRITISH "TEXTILE MANUFACTURER"

TEXTILE RESEARCH JOURNAL

VOL. XX, NO. 5 May, 1950

Mechanism of Microbiological Decomposition of Cellulose*

R. G. H. Siu

Contribution of the Quartermaster General Laboratories, Philadelphia, Pennsylvania; and the Farlow Herbarium, Harvard University, Cambridge, Massachusetts

Abstract

Knowledge of the mechanism of microbial action on cellulose is important for the solution of problems not only in textile technology but also in agronomy, plant pathology, bacteriology, mycology, medicine, geology, and the wood, paper, photographic, and allied industries.

Our present picture concerning the mode of breakdown of the cotton fiber is as follows: Upon germination, the microorganism secretes enzymes which first digest the cuticle. The hyphae of fungi then penetrate the secondary cellulosic wall directly into the lumen, where profuse growth ensues. Bacteria, on the other hand, adhere to the outer surface and pit their way inward. In both cases, digestion of cellulose occurs at the point of immediate contact with the organism.

In submicroscopic regions of the fiber, the amorphous cellulose is more rapidly attacked than the crystalline. The native cellulose molecule is first converted by an enzyme (called cellulase by the author) into a linear polysaccharide. Another enzyme then hydrolyzes the linear chain directly into glucose, without the intermediary of cellobiose. The proposed mechanism differs significantly from that of Pringsheim and of Winogradsky.

Based on this picture, topochemical modification appears to be very promising as a means of imparting simultaneously two or more properties to cotton fabrics, one of which is mildew-proofness.

WE in the textile industry are interested in the breakdown of cellulose by microorganisms from a rather restricted viewpoint. We look upon this process as an undesirable reaction because it brings about an annual damage to cotton textiles which amounts to hundreds of millions of dollars. And we want to prevent it.

Actually, for a broader biological point of view, the phenomenon is not only desirable but vital. It plays one of the most important roles in maintaining the carbon cycle in nature, without which life on this planet as we know it could hardly survive. The formation of humus as a result of microbial action on

* Presented before the 20th Annual Meeting of Textile Research Institute, Waldorf-Astoria Hotel, New York City, December 1, 1949.

plant remains in the soil is certainly of prime importance in increasing soil fertility. Furthermore, were it not for the vigorous microbiological activity in sewage disposal and garbage dumps, the problem of elimination of waste from modern industrial cities would be insoluble indeed.

The reaction is also of interest to investigators in scientific disciplines other than textile technology. The plant pathologist, for example, long concerned with the way in which pathogenic bacteria and fungi penetrate cell walls, is awaiting the clarification of the underlying mechanism of the microbiological breakdown of cellulose. Our associates in the wood, paper, photographic, plastic, and allied industries are as economically involved as we in this problem and are looking at it from the same angle of prevention.

Even the geologist and the medical scientist have shown deep cognizance of it from time to time. The former is puzzled over the contribution of microorganisms in the formation of petroleum and paleozoic coals from organic sediments. The latter, in the prescribing of cellulose derivatives as aids for intestinal disorders, is keenly interested in any possible action of intestinal microorganisms on such substances.

Despite the fact that all of these varied fields of science and technology are considerably interested in solving this riddle, the most vigorous contributors to the subject during the past decade have been those connected with the textile industry. So often the textile industry has been accused of scientific parasitism. It has been charged with adapting and exploiting knowledge unearthed by others without replenishing the well of scientific information so that others may benefit in turn. It is fitting to point out, therefore, that this fundamental problem of microbial action on cellulose is certainly one in which the textile industry is providing the greatest impetus today. Many other scientific and technological disciplines will benefit from its contributions.

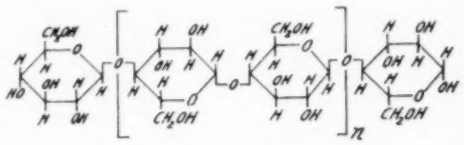
This discussion will deal with present-day thinking on the manner in which microorganisms breakdown the cotton fiber, the successive steps through which the cellulose molecule is converted as it is solubilized and used as food by the microorganism, and, finally, the practical implications of these basic interpretations.

It may be well to point out that most of the questions presented below are new and controversial. Some have not been published before and only a few have been proved beyond a shadow of a doubt. I believe, however, that there are sufficient data which can be pulled together to give a satisfactory tentative picture and a very useful guide for future thinking.

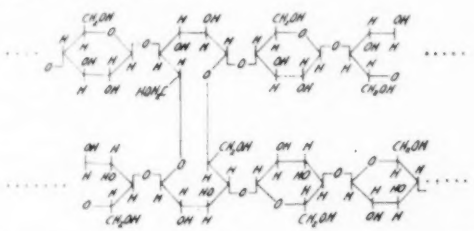
Structure of Cotton Fiber

The cotton fiber is a twisted ribbon about an inch long and a thousandth of an inch in diameter. It is covered with a thin outer layer or cuticle and contains a large central canal running the length of the fiber. The thick secondary wall is made up of cellulose molecules arranged in various ways. In certain microscopic areas they are arranged in a more orderly and compact fashion. These are known as crystalline areas. In other places the molecules are more randomly distributed. These are called amorphous areas.

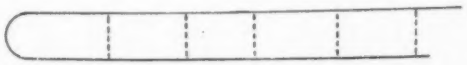
The chemical picture for the cellulose molecule is conventionally drawn as follows:



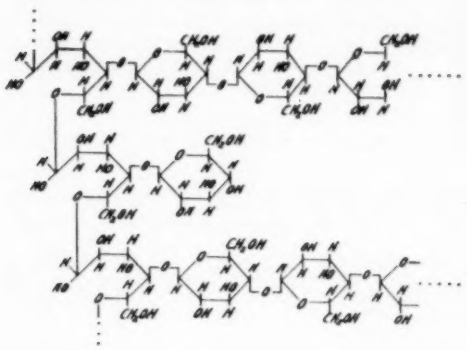
This structure, involving a long chain of 3,000 or more anhydroglucose units joined together by β-glycosidic linkages, is at least 99% correct. There seems to be some argument, however, over the remaining 1%. Hess and Steurer [9] suggested cross-linkages involving 12-membered rings which are formed by the reunion of two open anhydroglucose units, namely:



Haworth [7] had previously proposed chemical bonds joining adjacent polysaccharide chains at definite intervals. His concept can be diagrammed as follows:



During the past few years Pacsu [18] has put forth the concept of hemiacetal cross-linkages:



Despite disagreements over the precise nature of the cross-linkages, these and other workers [5, 12, 17] all seem to agree that in native cellulose there are present in small amounts chemical bonds other than the β-glycosidic type which serve to join the linear chains together.

Histological Penetration of Fiber by Microorganism

When the fungus or bacterium lands on the cotton fiber it begins to germinate in the presence of adequate moisture and favorable temperature. The first thing that happens is a digestion of the cuticle or outer non-cellulosic layer. Evidence for this statement can be deduced from results of microscopic examinations of cotton fibers swollen with sodium hydroxide and carbon bisulfide. The unattacked cotton fiber shows the well-known beading effect, which has been ascribed to the presence of the cuticle [1, 10]. In the case of a fiber which has been previously attacked by fungi or bacteria, however, there is no beading effect upon swelling with alkali and carbon bisulfide. This has been repeatedly observed both in our laboratories and in others [11, 28]. Such observations suggest an absence of the cuticle as a result of its digestion by microorganisms.

The next step involves the degradation of the cellulosic portions of the fiber itself. Here, the attack is not a generalized one as in the case of the cuticle. It is more localized. The fungus or bacterium apparently attacks the cellulosic wall at the point of immediate contact. Figure 1 illustrates the way in which fungi attack the secondary wall. The photomicrograph itself does not show the fungal hyphae, which have been dissolved during the preparation of the slide. By following the growth of the fungi with photomicrographs of this sort after various incubation periods, we can piece together what is happening. Upon germination, the fungal spore sends out a germ tube. The young hypha penetrates the cellulosic wall directly into the lumen. There the fungus grows profusely, digesting the fiber from the inside outward.

Some difference is observed in the attack of fibers by bacteria. Unlike fungi, bacteria do not grow in long filaments. Instead, they remain as small blobs of protoplasm, which multiply by pinching into two. Of necessity, therefore, bacteria are not able to penetrate the fiber by means of germ tubes as fungi are.

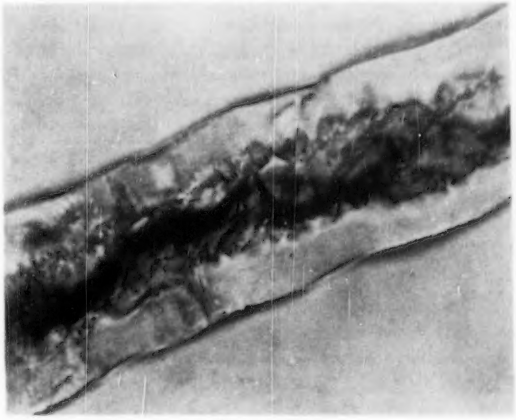


FIG. 1. *Photomicrograph of cotton fiber partially digested by the fungus,* Memnoniella echinata. *Magnification 800 ×. (Courtesy Miss Katherine Sanderson, Quartermaster General Laboratories.)*

FIG. 2. *Photomicrograph of cotton fiber partially digested by bacterium,* Sporocytophaga myxococcoides. *Magnification 300×. (Courtesy Dr. E. T. Reese, Quartermaster General Laboratories.)*

Instead, they etch the surface in the manner shown in Figure 2. The bacterium merely adheres to the fiber surface and pits away. The attack is from the outside inward.

Although the respective modes of histological attack by fungi and bacteria differ in the gross appearance, they appear similar in at least one important aspect. In both cases the digestion of cellulose occurs apparently only at the point of immediate contact between the organism and the cotton fiber. There appears to be no degradation of cellulose at a distance from this point. This concept of localized action based on sheer microscopic observations is supported by measurements on cuprammonium fluidity. Figure 3 shows Barghoorn's data [2] on the average cuprammonium fluidities of thousands of samples of cotton fabrics exposed in the sun and in the shade in Panama. The fluidity increases with time or with the loss in strength of the cloth exposed to the sun. This change of fluidity with photochemical degradation is a well-known phenomenon [8, 21].

However, the curve for deterioration in the shade, in which the causal agent was microbiological, is entirely different. In this case, there is no change in fluidity with time or loss of strength of the cloth. The same two types of curves were found by Dr. H. Rutherford (presented in Barghoorn's report) in the laboratory in working with hydrochloric acid and the cellulolytic fungus, *Chaetomium globosum*.

The results can be interpreted in the following way: Due to the highly localized action of microorganisms on the cloth, the cellulosic molecules at the point of immediate contact with the organism are rapidly digested before others are attacked. This means that the measurements on the fluidity of microbiologically degraded cloth do not reflect the fluidity of the digested cellulose molecules, which have been removed. Instead, the measurements are actually being made on the remaining molecules, which had not been attacked. Consequently, the observed fluidity remains the same.

Effect of Crystallinity and Orientation on Rate of Microbiological Action

From evidence available at the present time it seems quite certain that the amorphous areas in the cotton fiber are more rapidly attacked by microorganisms than the crystalline. This would appear reasonable even on *a priori* considerations. The looser-packed amorphous areas should allow for a more rapid accessibility by the enzymes secreted. Thus, ramie, a well-oriented and much more crystalline fiber than cotton, is attacked at a slower rate.

Further support can be gathered from the work of Karrer and associates [13]. They subjected two samples of viscose to the action of species of *Penicillium*. One sample was stretched-spun and the other was not. As shown by the values of tenacity and extension at break, the stretched-spun sample was much better oriented than the other. When both were subjected to attack by the mold, 31.7% of the

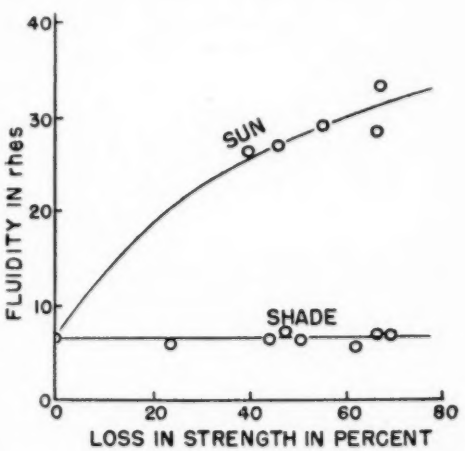


FIG. 3. *Cuprammonium fluidities of cotton fabrics exposed in Panama.*

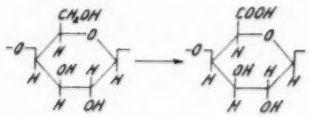
viscose spun without tension was digested as compared to only 9.1% for the other.

Similar results were obtained by Perlin and associates [19] with the cellulose-destroying bacterium, *Vibrio perimastix*. The rate of action on cellulose was accelerated when the amorphous content was increased by treatment with sodium hydroxide.

Degradative Transformations of the Cellulose Molecule

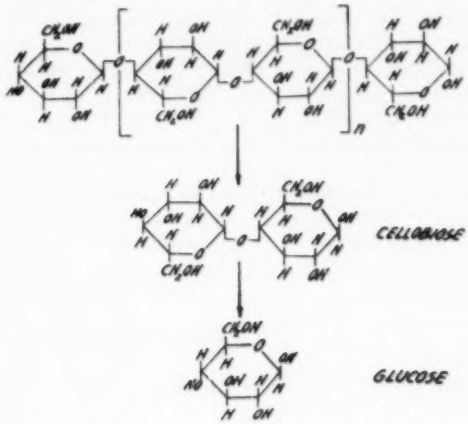
Since cellulose is insoluble in water and therefore cannot diffuse into the organism, it is logical to expect that the organism itself must secrete chemical substances which solubilize the cellulose. The water-soluble fragments can then diffuse into the cells and are used up as food. The chemical substances involved in carrying out this solubilization process are called enzymes. They are members of a large group of biological catalysts, which are responsible for the many biochemical reactions that go on in living systems.

Up to now, two theories have been proposed to explain the biochemical transformations undergone by the cellulose molecule. One is by Winogradsky [27], which is outlined as follows:



His theory stipulates that the first step involves the oxidation of the primary hydroxyl group to give 6-carboxycellulose. The latter is then presumably hydrolyzed to soluble compounds. The evidences against this explanation are so weighty that it must now be discarded. In the first place, although glucose has been isolated from the breakdown products of cellulose decomposition, the 6-carboxy derivative has not been reported. Furthermore, our preliminary studies show that 6-carboxycellulose is resistant to cellulose-decomposing microorganisms. It is hardly to be expected, therefore, that the first step in the conversion of cellulose to foodstuff would involve a resistant compound as an intermediate.

The second theory has been proposed by Pringsheim [20]. According to his explanation, the cellulose is first hydrolyzed to cellobiose by the enzyme cellulase. In the second step the cellobiose is split further to glucose by the enzyme cellobiase. The glucose is then used up as foodstuff by the microorganism. The sequence is written as follows:



Pringsheim's theory is in vogue today. It is freely quoted in reviews and textbooks as representing the most accurate picture available. Our recent findings on the subject, however, strongly suggest that his theory will have to be modified drastically [22].

There is no question that glucose is one of the main breakdown products. This sugar has been identified in the cultures growing on cellulose [20]. We have conducted quantitative studies [23] showing that it is the main product resulting from the action of enzymes from cellulolytic fungi.

There is also no question regarding the general pattern in which the glucose is digested within the cell. The biochemical pathway of glucose metabolism is similar to that in other living systems, as diagrammed in Figure 4.

We need not go into the voluminous data which have been gathered by many biochemists in piecing together this reliable metabolic picture for glucose. The reader is referred to other excellent reviews on the subject [14-16]. What especially concerns us here are the successive steps that take place between native cellulose and glucose.

Our first departure from Pringsheim's theory is the contention that cellobiose is not a necessary intermediary. This we base on the fact that we have obtained enzymic preparations which are capable of converting cellulose to glucose but not cellobiose to glucose. These preparations were obtained from microorganisms growing on cellulose. As a matter

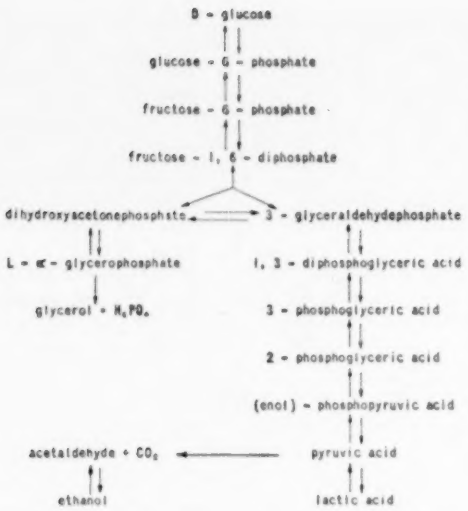


FIG. 4. *Glucose metabolic cycle in living systems.*

of fact, Grassmann and associates [6] have recorded comparable data in the literature, although they had not realized their theoretical significance. It hardly seems likely to us, therefore, that cellobiose is an intermediary when it cannot be hydrolyzed by the same enzymic preparation that can convert cellulose to glucose.

The next question involves the transformation of the insoluble native cellulose to the soluble sugar, glucose, without passing through cellobiose, of course. So far we have evidence to show that there are at least two separate steps concerned in the solubilization of native cellulose. Briefly, the conclusion is based on the following line of reasoning: It is known that organisms which are capable of digesting native cellulose are also capable of digesting the glycosidic linkages of partially substituted carboxymethyl-cellulose [26]. It can be assumed therefore that the enzyme that hydrolyzes the glycosidic bond in cellulose also attacks the same bond in the carboxymethyl-cellulose sample. Dr. E. T. Reese in our laboratories has observed quite a few species of fungi and bacteria which cannot attack native cellulose but yet are capable of hydrolyzing partially substituted carboxymethyl-cellulose. Finally, there are species which cannot attack either compound. These data are in harmony with early preliminary work of Seilliere [24], who claimed that snails are capable of digesting reprecipitated cellulose but not native cellulose.

The two enzymic steps involved in the microbiological solubilization of native cellulose are diagrammed as follows:

native cellulose
↓
linear polysaccharide chain
↓
glucose

The same data may be regarded as biological or biochemical evidence, one may say, of the presence of linkages other than β-1,4-glycosidic bond in the native cellulose.

At the present time we know nothing about the mechanism of the first step, which is responsible for the conversion of native cellulose to the linear chain. We suggest reserving the term "cellulase" for the enzyme which causes the initial degradative change in native cellulose. Regarding the second step, however, we have been able to uncover some interesting data. We believe that the organism need not neces-

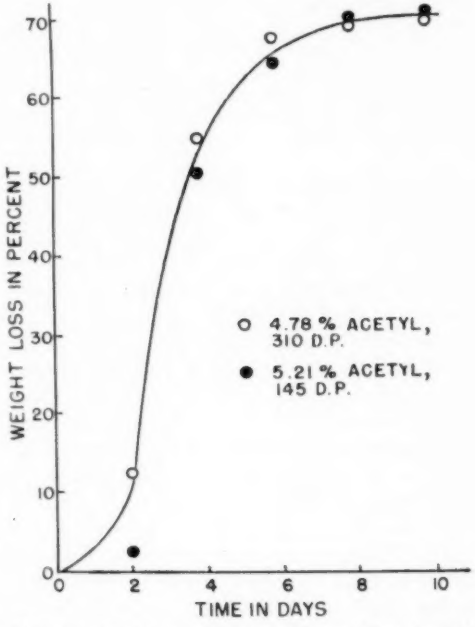


FIG. 5. *Growth of cellulolytic fungus,* Myrothecium verrucaria, *on partially substituted cellulose acetates.*

sarily proceed at one end of the polysaccharide chain and digest off one anhydroglucose unit after another, according to Clayson's belief [3]. Instead, the organism is able to initiate attack on any glycosidic linkage along the chain. Illustrative supporting data are presented in Figure 5.

The substrates used were partially acetylated cellulose with different degrees of polymerization or chain lengths. The samples were prepared in phosphoric acid solution so that acetylation occurred more or less at random along the polysaccharide chain. The high degree of breakdown indicates that the internal glycosidic linkages are susceptible to microorganisms. Whether or not the terminal or the internal linkages are attacked at greater rates is not clear at the present time, although we had once speculated on rather flimsy evidence that they were equally susceptible [26].

As far as cellulose derivatives are concerned, complete resistance to microorganisms will be exhibited only when there is one or more substituents on every anhydroglucose unit. The glycosidic linkages adjacent to any unsubstituted anhydroglucose unit seem to be readily attacked. The degree of resistance does not appear to be affected by the nature of the substituent. The list of derivatives tested so far include methyl, ethyl, tosyl, iodotosyl, carboxy, carboxymethyl, and acetyl compounds [26].

Practical Implications

The mechanism proposed is admittedly a tentative picture. Even in its crude state, however, it has considerable practical implications [25]. Two aspects of the whole picture are important in this respect.

In the first place, since the attack by microorganisms is a localized one, it should be theoretically possible to make the cotton fiber mildewproof by coating it with a thin layer of resistant material. We can produce this protective barrier by a surface chemical reaction on the fiber. In the second place, since the degree of resistance is not determined by the nature of the substituent, it should be possible to use reacting groups which by their very nature possess other desirable properties, such as flameproofness, water-repellency, and photochemical stability. In plant applications it is conceivable that the reaction can be caried out on the finished fabric in the piece by passing it through a reagent bath and a baking oven to ensure completion of the reaction [4].

The resulting mildew-resistant treatment should be resistant to leaching, inasmuch as it is an integral part of the fiber. It is not physically adsorbed on the fabric as in the case of fungicides. The treatment should also be non-toxic to human beings. The cellulose derivatives resulting from such methods are not toxic by themselves, and are not water-soluble, as poisons must be in order to exhibit toxicity. This, of course, is a great advantage in the case of fabrics which are to be used in contact with humans. Finally, it will also provide an opportunity to impart other highly desirable properties to the cloth at no extra cost.

So far, all experiments on this so-called topochemical modification of fabrics have borne out the predictions. The studies have so far been restricted to laboratory scale, however. But it is hoped that the idea will gain momentum with time so that before long such treatments will be standard mill practice.

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Wax Content as Related to Surface Area of Cotton Fibers

Paul B. Marsh, Henry D. Barker, Thomas Kerr, and Mary L. Butler*

Abstract

Among a group of 62 essentially unweathered fiber samples from American upland cotton plants of different inherent and environmental background, percent wax was found to be rather closely proportional to surface area per unit weight of fiber as determined by the air-flow method of Sullivan and Hertel [22]. The weight of wax per unit of fiber surface was thus a relatively constant quantity, although not an absolutely constant one, among these samples. Results of melting-point determinations did not suggest the probability of important qualitative differences in wax composition at the time of boll opening among the several American upland cottons studied, the sole exception encountered being the high-melting-point wax from the green-lint high-wax cotton previously reported by Conrad [6].

In a group of twelve Sea Island hybrids, both the amount of wax per unit fiber surface and the wax melting points were similar to the results obtained for American upland types. Five Indian cottons all had very low wax percentages; their surface areas were below the normal range of the method for surface-area determination.

Weathering of cotton in the field before harvest resulted in higher wax contents as measured by the Conrad [7] method and lower wax melting points than were found for similar but unweathered samples. Further work is required to indicate the nature of the causal factors involved in this change which occurs during weathering.

ALTHOUGH several investigations have been carried out on the chemical nature of cotton wax [3, 5, 6, 11, 12, 17, 23] and on methods for its quantitative determination [4, 7, 10, 14, 15], exceedingly little has been published in an attempt to relate quantitative or qualitative *differences* in wax among different cotton samples to the inherent or environmental background of the plant, to other fiber properties, or to the actual use value of cotton. For cotton varieties of current agricultural importance in the United States this is particularly true, almost no information on the subject appearing to have been printed. The subject of differences in wax among cottons, however, is of interest for several practical reasons. Cotton wax has long been generally believed to influence spinning performance and abrasion-resistance of cotton and clearly is a primary factor in determining wettability of the fiber. Certain agricultural investigators have also considered that

* Bureau of Plant Industry, Soils, and Agricultural Engineering, Agricultural Research Administration, U. S. Department of Agriculture, Beltsville, Md.

the fiber's resistance to damage during weathering before harvest and its ease of cleaning might likewise be related to wax content. In addition, wax is of practical concern as one of the more difficult of the secondary fiber constituents to remove in kiering and bleaching [14].

Since several properties of practical importance which have been attributed to cotton wax are exerted at the fiber surface and since the material commonly has been considered to be primarily, if not entirely, a surface constituent, it seemed logical that a quantitative study of cotton wax should be in some way related to information on the surface of the fiber. The availability of facilities for determining surface area per unit weight of fiber at the University of Tennessee Fiber Laboratory by the procedure of Sullivan and Hertel [22] suggested a method of experimental approach to percent wax-surface area comparisons and to an investigation of wax content on a weight-per-unit-surface-area basis rather than simply as percent wax.

The original purpose of the present investigation was to determine whether inherent or environmental differences in wax content of large enough magnitude to be of probable practical significance exist among American upland cottons. For comparative purposes several Sea Island and a few Indian cottons were also tested. While the information obtained to date is not comprehensive enough to yield any final answer to the original question, it has become clearly apparent that, in general, differences in percent wax are in large measure reflections of differences in surface area per unit fiber weight among cottons, and the data now available are presented primarily from the standpoint of demonstrating the relationship between these two fiber properties. The wax-surface relationship, however, is not entirely free of complicating factors, since an apparent increase in wax content as measured by the Conrad method has been found to occur, at least under some circumstances, during weathering of the fiber in the field before harvest. This phenomenon is in need of further investigation.

Methods

Most of the samples (*i.e.*, samples 1–74, Table II) were collected within a few days after boll opening from plants grown at the several U.S.D.A. field stations at the locations noted in Table II. Most of the varieties of currently major agricultural importance in the United States are represented among the samples tested. Samples 1–62 were 60-boll samples and because of this relatively small size or for other reasons the results here presented may not all be accurately representative of the fiber from the entire planting of each individual variety at the different locations. In general, however, the larger of the varietally associated differences in surface area which may be noted in the data are in accord with much previous information, and it is believed that few if any samples seriously atypical of the varieties as named are included.

Wax analyses were carried out in duplicate by the method of Conrad [7], with the exception of samples 78 and 81, on which single analyses only were run. This procedure involves the extraction of the wax from the fiber in a Soxhlet apparatus with hot ethyl alcohol. Information in the original description of the method [7] and in a later paper by Kettering, Goldthwait, and Kraemer [15] indicates that with hot ethyl alcohol [7, 15] or hot isopropyl alcohol [15] the wax extraction is more rapid and reaches a complete or nearly complete maximum more quickly than is the case with chloroform or benzene; the opinion is expressed [7, 14, 15] that these alcohols are for this and/or other reasons more satisfactory than any of the several other organic solvents which have been used for cotton wax analyses by earlier workers. The nonwaxy constituents extracted from the fiber along with the wax in the Conrad [7] method remain with the alcohol-water layer when chloroform and water are added to the alcoholic extract in a separatory funnel, and the wax passes into the chloroform layer, which is washed with water and later heated on a steam bath to evaporate off the chloroform. In the present work the solvent evaporation was carried out in 50-cc. weighing bottles, which were then cooled to

TABLE I. ANALYTICAL RESULTS FOR PERCENT WAX OF RAW COTTON AS RELATED TO THE TOTAL PERIOD OF EXTRACTION, USING THE METHOD OF CONRAD [7]

Variety	Sample No.	Total percent wax extracted* 5 hrs.	6 hrs.	7 hrs.	8 hrs.
Acala 5-12-7-6	1	.728	.728	.730	.728
	2	.735	.736	.735	.735
Rowden 41B	1	.465	.469	.469	.469
	2	.462	.462	.462	.462
Coker Wilds	1	.700	.701	.701	.701
	2	.706	.707	.707	.707

* Each sample was extracted for 5 hrs. and then for three successive additional 1-hr. periods with fresh alcohol.

TABLE II. RESULTS OF DETERMINATIONS OF PERCENT WAX, SURFACE AREA PER UNIT WEIGHT, AND WAX MELTING POINT FOR VARIOUS COTTONS

Beltsville sample no.	Variety, strain, or other designation	Location of growth	Year of growth	Percent wax	Surface area ($cm.^2/mg.$)	Wax per unit surface (gammas per $cm.^2$)	Melting point (°C)
1	Acala 1517W	Florence, S. C.	1948	.623 ± .002	2.86	2.18	72–78
2	Acala 4-42	Florence, S. C.	1948	.597 ± .001	2.50	2.39	70–76
3	Acala 5675	Florence, S. C.	1948	.587 ± .002	2.55	2.30	—
4	Cobal T-416	Florence, S. C.	1948	.494 ± .010	2.35	2.10	—
5	Coker 100W	Florence, S. C.	1948	.591 ± .001	2.40	2.46	70–75
6	CSS-9	Florence, S. C.	1948	.533 ± .007	2.56	2.08	—
7	Empire WR	Florence, S. C.	1948	.573 ± .004	2.69	2.13	—
8	Hybrid 1282	Florence, S. C.	1948	.600 ± .006	2.59	2.32	—
9	Pandora	Florence, S. C.	1948	.557 ± 0	2.43	2.29	72–76
10	Sealand 542	Florence, S. C.	1948	.698 ± .004	3.10	2.25	71–76
11	Smith's 78	Florence, S. C.	1948	.544 ± .004	2.56	2.12	—
12	Stonewilt	Florence, S. C.	1948	.547 ± .002	2.38	2.30	—
13	Watson's Mebane	Florence, S. C.	1948	.588 ± .004	2.29	2.57	71–75
14	White Gold	Florence, S. C.	1948	.575 ± .004	2.76	2.08	72–77
15	364–367	Florence, S. C.	1948	.467 ± .002	2.21	2.11	—
16	382–384	Florence, S. C.	1948	.575 ± .004	2.74	2.14	—
17	385–387	Florence, S. C.	1948	.568 ± .003	2.57	2.21	—
18	397–399	Florence, S. C.	1948	.746 ± .006	3.09	2.41	—
19	424–429	Florence, S. C.	1948	.615 ± .010	2.51	2.45	—
20	441–448	Florence, S. C.	1948	.949 ± .002	3.74	2.54	—
21	456–465	Florence, S. C.	1948	.741 ± .008	3.01	2.46	—
22	545–550	Florence, S. C.	1948	.670 ± .008	2.77	2.42	—
23	551–555	Florence, S. C.	1948	.630 ± .001	2.81	2.24	—
24	30 H	Shafter, Cal.	1947	.628 ± .002	2.74	2.29	—
25	51 H	Shafter, Cal.	1947	.579 ± .004	2.43	2.38	—
26	52 H	Shafter, Cal.	1947	.592 ± .002	2.50	2.37	—
27	55 H	Shafter, Cal.	1947	.546 ± .002	2.31	2.36	—
28	60 H	Shafter, Cal.	1947	.561 ± .002	2.61	2.15	74–77
29	65 H	Shafter, Cal.	1947	.509 ± .004	2.35	2.17	71–73
30	81 H	Shafter, Cal.	1947	.637 ± .005	2.71	2.35	71–74
31	29 L	Shafter, Cal.	1947	.568 ± .003	2.51	2.26	73–76
32	33 L	Shafter, Cal.	1947	.559 ± .001	2.50	2.24	72–75
33	34 L	Shafter, Cal.	1947	.605 ± .003	2.48	2.44	70–73
34	46 L	Shafter, Cal.	1947	.523 ± .006	2.38	2.20	—
35	47 L	Shafter, Cal.	1947	.581 ± .006	2.41	2.41	—
36	85 L	Shafter, Cal.	1947	.560 ± .004	2.33	2.40	—
37	116 L	Shafter, Cal.	1947	.647 ± .008	2.92	2.22	—
38	Acala 5-12-7-6-WX	Shafter, Cal.	1947	.755 ± .002	2.93	2.58	67–71
39	Rowden 41B	Florence, S. C.	1948	.497 ± .008	2.13	2.33	74–78
40	Rowden 41B	Stoneville, Miss.	1948	.528 ± .004	2.17	2.43	72–75
41	Rowden 41B	Shafter, Cal.	1948	.587 ± .003	2.40	2.44	70–74
42	Rowden 41B	Statesville, N. C.	1948	.646 ± .012	2.54	2.54	69–75

room temperature in a desiccator over Drierite before weighing. The ethyl alcohol maintained a temperature in the Soxhlet extracting chambers of 74°C or higher. Siphoning of the solvent occurred with a frequency of once in every 3–4 min., and a rather complete removal of the solvent with each siphoning was accomplished as a result of the use of thimbles (coarse Alundum, RA 98) with 12 holes of approximately 0.06-in. diameter bored through them. The fiber was cleaned free of trash, motes, and other extraneous material and was thoroughly mixed by hand before analysis, an 8-g. sample being used for each extraction. All results are expressed on the basis of oven-dry weight of the raw fiber.

Completeness of wax extraction, or at least the maximum amount of wax extractable by the method used, was known to have been attained within the specified 6-hr. extraction period [7] (conclusion based on the test results shown in Table I). The absence of significant residues after solvent evaporation for the later extractions indicates also the absence of any measurable error which might have

TABLE II—*Continued*

Beltsville sample no.	Variety, strain, or other designation	Location of growth	Year of growth	Percent wax	Surface area (cm.²/mg.)	Wax per unit surface (gammas per cm.²)	Melting point (°C)
43	Rowden 41B	State Coll., N. M.	1948	.638 ± .014	2.50	2.55	70–75
44	Deltapine 15	Florence, S. C.	1948	.537 ± .005	2.45	2.19	71–76
45	Deltapine 15	Stoneville, Miss.	1948	.598 ± .008	2.59	2.31	70–74
46	Deltapine 15	Shafter, Cal.	1948	.704 ± .010	2.97	2.37	68–72
47	Deltapine 15	Statesville, N. C.	1948	.852 ± .004	3.32	2.57	70–74
48	Deltapine 15	State Coll., N. M.	1948	.787 ± .007	2.97	2.65	68–73
49	Stoneville 2B	Florence, S. C.	1948	.631 ± .003	2.70	2.34	72–77
50	Stoneville 2B	Stoneville, Miss.	1948	.702 ± .007	2.84	2.47	70–73
51	Stoneville 2B	Shafter, Cal.	1948	.792 ± .008	3.21	2.47	68–75
52	Stoneville 2B	Statesville, N. C.	1948	.875 ± .009	3.44	2.54	69–73
53	Stoneville 2B	State Coll., N. M.	1948	.796 ± .002	3.02	2.64	68–74
54	Coker Wilds	Florence, S. C.	1948	.645 ± .002	2.93	2.20	73–77
55	Coker Wilds	Stoneville, Miss.	1948	.761 ± .002	3.05	2.50	70–73
56	Coker Wilds	Shafter, Cal.	1948	.893 ± .010	3.27	2.73	70–74
57	Coker Wilds	Statesville, N. C.	1948	.951 ± .009	3.92	2.43	72–77
58	Coker Wilds	State Coll., N. M.	1948	.908 ± .004	3.31	2.74	71–74
59	Acala 4–42 (chalazal)	Florence, S. C.	1948	.672 ± .004	2.87	2.34	68–75
60	Acala 4–42 (mid-seed)	Florence, S. C.	1948	.538 ± .002	2.14	2.51	64–74
61	Coker 100W (chalazal)	Florence, S. C.	1948	.738 ± .001	2.92	2.53	67–73
62	Coker 100W (mid-seed)	Florence, S. C.	1948	.524 ± .002	2.15	2.44	67–73
63	$(PY)_7$ 1083*	Tifton, Ga.	1948	.717 ± .026	2.78	2.58	72–76
64	$(PY)_7$ 1084	Tifton, Ga.	1948	.765 ± .006	3.19	2.40	74–78
65	$(WY)_7$ 1103	Tifton, Ga.	1948	.647 ± .006	2.68	2.41	75–78
66	$(WY)_7$ 1107	Tifton, Ga.	1948	.664 ± .027	2.84	2.34	75–78
67	$Y(WV)_5$ 1140	Tifton, Ga.	1948	.741 ± .008	3.05	2.43	72–77
68	$Y(ZV)_5$ 1142	Tifton, Ga.	1948	.704 ± .007	3.06	2.30	72–76
69	$Y(ZV)_5$ 1143	Tifton, Ga.	1948	.758 ± .002	3.09	2.45	72–75
70	$(YR)_7$ 1174	Tifton, Ga.	1948	.648 ± .004	2.75	2.36	75–78
71	$(YR : TZ)_5$ 1128	Tifton, Ga.	1948	.730 ± .028	2.93	2.49	72–77
72	$(YR : TZ)_5$ 1129	Tifton, Ga.	1948	.706 ± .007	2.91	2.43	73–77
73	$Y(TZRV)_5$ 1089	Tifton, Ga.	1948	.764 ± .020	3.16	2.42	70–74
74	$Y(TZRV)_5$ 1167	Tifton, Ga.	1948	.641 ± .004	3.00	2.14	73–77
75	Karnak	Unknown	Unknown	.641 ± .004	3.21	2.00	75–78
76	Tanguis	Unknown	Unknown	.665 ± .004	2.34	2.84	70–76
77	Garo Hill-R	Raleigh, N. C.	1940	.205 ± .004	1.30	1.58	65–72
78	Garo Hill-S	Stoneville, Miss.	1948	.190	1.28	1.48	65–71
79	"Indian cotton" (PI-180394)	India	Unknown	.260 ± .008	1.52	1.71	68–70
80	"Indian cotton" (PI-18117)	India	Unknown	.287 ± .001	1.51	1.90	70.5–72.5
81	"Indian cotton" (PI-180363)	India	Unknown	.219	1.32	1.66	65–71

* Among the Sea Island hybrids, listed as samples 63–74, the symbols P, R, T, V, W, and Z represent the varieties Montserrat, Porto Rican, Westberry, Bleak Hall, Concord, and Seabrook, respectively.

arisen from the presence of dissolved solid impurities in the reagents. The average deviation from the mean of duplicate wax analyses, as calculated from the data of Table II, was .8% of the mean value.

Determinations of surface per unit fiber weight were carried out by the method of Sullivan and Hertel [22] at the University of Tennessee Fiber Laboratory on 200-mg. subsamples from the cleaned and well-mixed large samples from which were also taken the samples for wax analyses. The surface area values for Figure 2 were averages of determinations on 2 or, more commonly, on 4 individual subsamples, the average deviation from the mean being 1.73%.

The general features of construction and the method of use of the arealometer, as employed in the determinations reported in the present paper, may be described as follows. The instrument is essentially a pneumatic Wheatstone bridge, two arms of which have equal resistances, while the other two

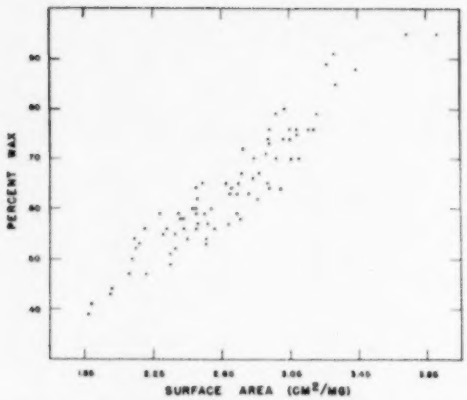

FIG. 1. *Percent wax and surface area per mg. of 74 samples of cotton.* ● = *American upland cottons.* × = *Sea Island cottons. Description of samples and numerical data are listed as items 1 to 74 in Table II. Correlation coefficient, r, for percent wax vs. surface area* = .94.

arms consist of the sample of cotton being tested and a standard resistance. Air is supplied to the bridge from a weighted bellows which is actuated by an aspirator bulb. The cotton is held in a cylindrical chamber with a cross-sectional area of .082 sq. in. and with a perforated end, and is compressed by a perforated plunger driven by a screw. A liquid manometer connected across the equal arms indicates when the cotton is compressed to such an extent that it offers a resistance to the flow of air equal to that offered by the standard known resistance (2,596 dynes/cm.²/cc./sec.). With air flowing through the instrument, the resistances are equal when the liquid in the manometer columns is level. A dial operated by the compressing plunger measures the length of the compressed sample, which in turn is a measure of its specific surface in cm.²/mg.

The calibration curve for the instrument was drawn by plotting arealometer dial reading (length of sample plug) *vs.* cm.²/mg. as obtained from microscope measurements made on about 10 standard cottons. Each point on the calibration curve represented approximately 1,000 fibers whose cross sections were drawn from a microscope slide by means of a cameralucida. The areas of the fiber cross sections were measured with a planimeter and their perimeters were measured with a map measurer. The ratio of perimeter area is equal to the surface per unit volume (cm.²/cm.³), which in turn can be expressed as cm.²/g. after dividing by the density. On a well-mixed standard cotton in sliver form, coefficients of variability have been obtained of from 1.5% to 1.8%. Raw cottons which have not been homogenized give a slightly higher coefficient of variability, ranging between 1.8% and 3.0%.

Melting points were determined on a Fisher-Johns electrically heated aluminum block melting-point apparatus, with the sample being placed on the surface of circular No. 2 microscope cover slips. The material tested was in each case a subsample taken from the wax obtained in the quantitative determinations. The individual melting points covered a range of from a few to several degrees and provide, therefore, only an approximate comparative index of differences in composition among the different cotton waxes. Because of the existence of a time lag between the temperature of the block and the recording of the block temperature by the thermometer in this method, it is necessary to raise the temperature of the block very slowly. This precaution was taken.

Experimental Results

Results of wax and surface-area analyses of fiber picked promptly after boll opening from a number of different varieties and strains of American upland cotton at several locations and from several Sea Island cottons grown at Tifton, Georgia, are shown in Figure 1. Sample descriptions and numerical results corresponding to the points in the figure are recorded in the first 74 entries of Table II. The *r* value for correlation between surface area and percent wax for the data of Figure 1 was calculated to be .94. Data on green-lint cotton, an unusual American upland type of no present agricultural importance and known previously to have very high wax content [6], are not included in this calculation but are discussed later (page 293).

Percent wax tends strongly to be correlated with fiber surface area per unit weight regardless of whether variations in the latter quantity are of varietal or environmental origin. Data from samples 39 to 58 are graphed to bring out this fact in Figure 2. It is clearly apparent from the figure that Rowden 41B is lower than the other 4 varieties represented in Figure 2 in percent wax and in surface area, and that Coker Wilds is the highest of the 4 in both properties. It may also be seen that the cotton grown at

Florence, S. C., was lower than those from the other 4 locations represented in percent wax and in surface area and that, on the other hand, cotton from Statesville, N. C., was highest in both characters. Within the data for each variety, however, the ratio of wax to surface area exhibits a small but definite environmental trend. This trend may be seen by examining the figures for wax per unit surface area within the sample groups 39–43, 44–48, 49–53, and 54–58 in Table II. A trend of the magnitude of variation involved in these values could conceivably result from a variable amount of residual oil from the fiber lumen, which would be included in the "wax." Variation in apparent wax per unit surface area from this cause, however, would be expected to result in concurrent variation in wax melting points, a circumstance which, as may be seen from the melting-point data for samples 39–58 of Table II, did not occur. On the contrary, data on wax melting points for these and several other samples of American upland cottons, as recorded in Table II, provide no indication of any major differences in the original composition of the wax from this or any other cause among the unweathered American upland samples studied, the sole exception being the previously known high-melting-point wax of green-lint cotton.

Samples 24–30 were experimental strains which had been selected by feel and appearance prior to analysis as cottons believed to be "waxy" or "high in wax," whereas samples 31–37 were similarly selected as cottons "low in wax." The cause of the apparent difference in "waxiness" remains unknown, since test results failed to reveal any significant difference between the two groups in wax content, wax melting points, or surface areas. In some cases estimates of "feel" may possibly be influenced by the observer's impression of the luster of the fiber. Adderley [1] has shown that this property is closely connected with the shape of the cross sections of the fibers, cotton fibers which tend to be circular in cross section having a higher luster than those whose sections are flattened.

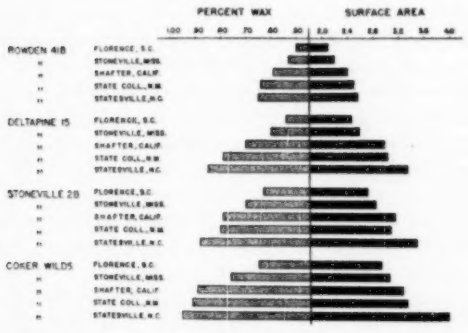

FIG. 2. *Percent wax and surface area per unit weight (cm.²/mg.) for 4 cottons grown in 5 locations in 1948. Data indicate correlation of percent wax with surface area regardless of whether variations in surface area are of varietal or environmental origin, variety Rowden and location Florence, S. C., being lowest in both properties and variety Coker Wilds and location Statesville, N. C., highest in both.*

Fibers from the chalazal, or rounded end of the seed, are known to be thinner-walled and consequently higher in surface area per unit weight than fibers from other locations on the seed. They might be expected, therefore, to yield a higher percentage of wax, an expectation which is, in fact, fulfilled by the experimental data (Table II, samples 59–62).

A few analyses have been made for comparative purposes on cottons outside of the American upland group. The results obtained for percent wax and surface area with such cottons are shown under the entries for samples 63 to 81 in Table II. The Sea Island hybrids (samples 63 to 74) showed, in general, wax percentages and melting points very similar to those found for American upland types. With the few Indian cottons tested (samples 77 to 81) the values for surface area were below the normal range of operation and calibration of the arealometer and the data for surface area and wax per unit surface area may therefore perhaps not be highly accurate. The very low wax percentages, however, clearly reflect the coarse nature of the fiber.

Four samples of green-lint cotton of the same general genetic background as the samples tested by Conrad [6] yielded values of 9.56, 7.63, 7.67, and 8.99 for percent wax, with melting points of 91–96°C, 91–95.5°C, 90–95°C, and 90–97°C, respectively. The wax in this cotton is believed to be located, at least in part, within the fiber wall.

Weathering of cotton in the field before harvest has been found to result in higher wax percentages as measured by the Conrad method [7] than were found for unweathered fiber from the same plants, and also in a distinct lowering of the melting point of the wax obtained. Data on the subject are given in Table III. The effects of weathering as shown in this table could presumably be due to the influence of sunlight

TABLE III. INFLUENCE OF WEATHERING ON PERCENT WAX AS MEASURED BY THE CONRAD [7] METHOD OF ANALYSIS AND ON WAX MELTING POINTS
(SAMPLES FROM CROP OF 1948; WEATHERED FIBER EXPOSED 11 WEEKS IN THE FIELD BEFORE HARVEST)

Test series	Location of growth	Variety	Condition of fiber	Percent wax	Surface area (cm.²/mg.)	Wax per unit surface (gammas /cm.²)	Melting point (°C)
1	Florence, S. C.	Coker Wilds	Unweathered	.645	2.92	2.21	73–77
			Weathered	.823	2.87	2.87	65–72
	Florence, S. C.	Stoneville 2B	Unweathered	.631	2.70	2.34	72–77
			Weathered	.701	2.57	2.73	65–73
	Florence, S. C.	Deltapine 15	Unweathered	.537	2.46	2.18	71–76
			Weathered	.782	2.43	3.22	65–71
1	Shafter, Cal.	Coker Wilds	Unweathered	.893	3.27	2.73	70–74
			Weathered	.945	3.08	3.07	64–71
	Shafter, Cal.	Stoneville 2B	Unweathered	.792	3.21	2.47	68–75
			Weathered	.877	3.46	2.53	64–70
	Shafter, Cal.	Deltapine 15	Unweathered	.704	2.97	2.37	68–72
			Weathered	.826	2.98	2.77	66–70
2	Florence, S. C.	Coker Wilds	Unweathered	.686	2.58	2.66	69–75
			Weathered	.779	2.77	2.81	69–74
	Florence, S. C.	Stoneville 2B	Unweathered	.688	2.40	2.87	70–76
			Weathered	.732	2.56	2.86	70–78
	Florence, S. C.	Deltapine 15	Unweathered	.622	2.33	2.67	72–77
			Weathered	.855	2.12	4.03	63–70
2	Shafter, Cal.	Coker Wilds	Unweathered	.964	3.07	3.14	70–75
			Weathered	.978	3.13	3.12	65–70
	Shafter, Cal.	Stoneville 2B	Unweathered	.828	2.91	2.85	70–76
			Weathered	.884	3.22	2.75	66–71
	Shafter, Cal.	Deltapine 15	Unweathered	.799	2.80	2.85	70–76
			Weathered	.852	2.82	3.02	65–70

in degrading and making soluble in alcohol and chloroform materials which were originally in an insoluble state in the unweathered fiber. It is recognized, however, that other explanations are also possible and that the phenomenon has not yet been adequately studied from the standpoint of the causal factors involved. If in a series of samples of unknown variety and history the differences in wax per unit surface area of the magnitude of those recorded in Table II for unweathered samples 1–62 should exist and should have superimposed on them differences in apparent wax content as a result of differences in the degree of weathering, the percent wax-surface area correlation could conceivably be rather poor or even very poor.

Discussion

Shortly after the beginning of the present investigation, the authors' attention was called to a paper by Nanjundayya [19] in which a correlation had been found to exist between percent wax and surface area per unit weight among the members of a series of Indian cottons. In general, the results of the present paper, concerned primarily with American upland and Sea Island cottons, are in good accord with those of Nanjundayya [19] on Indian cottons in the sense of indicating a high degree of correlation between wax content and surface area. Differences between the test methods of Nanjundayya and those used in the present work, however, render quantitative comparisons between the results of the two investigations very difficult. While such differences are too numerous to discuss in detail, a few points may be mentioned to indicate the nature of the difficulty. Evidence presented by Kettering, Goldthwait, and Kraemer [15] suggests that Nanjundayya's [19] wax extractions, using a 4-hr. extraction period with benzene, were probably less complete than those reported here. Deviations of at least some samples from the ideal cross-sectional shape assumed in his calculations of fiber surface must also have occurred. As a qualification to the results here reported, it must also be stated, however, that air-flow methods

of determining fiber surface, while yielding results which are commonly reproducible with considerable accuracy on a given instrument, are generally admitted to be insufficiently standardized with respect to results on identical samples obtained by different instruments. The data for surface area as here reported are presumably moderately but probably not extremely accurate in an absolute sense.

Prior to the work of Nanjundayya [19] the general tendency for percent wax to increase with increase in fiber fineness among American upland cottons had been suggested by certain limited data involving a set of 5 cotton samples reported on in 1941 by Nickerson, Fontaine, and Leape [20]. These authors noted a tendency for percent wax to parallel fineness as measured in terms of length per unit fiber weight. Since the best present information indicates, however, that cotton wax resides in and is functional at the fiber surface, surface area per unit weight seems to be a more logical quantity with which to compare percent wax than does length per unit weight. It is clear from the data in other contributions from this Division [13, 21] that the mechanically determined or U.S.D.A. weight per inch, as used by Nickerson, Fontaine, and Leape [20], may justifiably be considered to be inversely proportional to surface area per unit weight only among fibers of equal perimeter; and that while perimeter is a relatively constant property among American upland cottons, it is by no means completely so, and cottons differing widely in perimeter from the average are known.

The possibility that cotton wax might not occur solely on the fiber surface but that at least part of it might be located within the fiber was advanced by Knecht and Streat [16], who found higher wax percentages for cotton if the fiber were acid-treated or ground before extraction and who regarded these results as evidence of the physical protection of wax from extracting fluids by the cellulosic fiber wall. In a further investigation of this phenomenon, Lesslie, Hagan, and Guthrie [18] have failed to confirm the occurrence of a consistent increase in hot-alcohol-extractable wax following grinding and have noted that the increase in "wax" content which occurs as a result of prior exposure of the fibers to the fumes of HCl is not associated with true wax but rather with the action of the acid on some water-soluble material or materials present in the fiber. Fargher, Hart, and Probert [9] and Fargher and Higginbotham [10] previously had also carried out experiments relating to the claims of Knecht and Streat [16] and had stated [10]: "There is no evidence, therefore, for the existence of relatively large amounts of wax and resin which are protected from attack by their position within the hair."

If wax were deposited as a component not only of the primary wall of the fiber but of the secondary wall as well, it would seem that, within a group of fiber samples from a given cotton variety, those sample with unusually thick walls, as evidenced by low values for surface area per unit weight, would have high "wax per unit surface." As may be seen from Table II (samples 39–58), the values for wax per unit surface area were actually slightly lower on such samples.

From his data on wax per unit surface area and a figure for the density of cotton wax, Nanjundayya [19] calculates a hypothetical value of approximately .015 micron for the thickness of the wax layer on the surface of Indian cottons and notes that this could account for only 1–3% of the thickness of the primary wall. He adds, however, the qualifying statement that ". . . it would be premature to speculate as to whether the wax is located at the surface as a homogeneous layer or the whole primary wall is impregnated with it." A value of .025 micron for thickness of the wax layer of American upland cottons may be calculated by the same method from the data of Figure 1. Since the primary wall is extremely thin in comparison with the secondary wall and is essentially proportional to the fiber surface, the data here presented do not appear to offer any evidence of whether the wax is only on the outer surface of the primary wall or is also within it.

The range of surface-area values for American upland cottons represented by the data of Figure 1 is not extremely large and both higher and lower values than those shown here have been noted at the Tennessee Fiber Laboratory. It seems highly probable, therefore, that the range of values for percent wax among American upland cottons could readily be extended beyond the limits here recorded by testing cottons with extremely large or small surface areas.

Of the various kinds of fatty substances extractable from cotton fiber by the Conrad method [7], some could conceivably exist within the protoplasm rather than on the cell surface and could thus be subject to metabolism or to translocation to and

from the cell. The relative constancy of the ratio of wax as determined by the Conrad method to surface area as here shown, however, suggests that such a labile protoplasmic wax fraction must be very small. Because of their labile nature such protoplasmic materials as sugars, proteins, amino acids, and mineral salts might be expected to bear a much less fixed relationship to fiber surface area than does wax. In this connection it may be noted that Eaton [8] reported a very low correlation between percent nitrogen and "fiber maturity."

From the data here presented, it seems apparent that differences in wax percent among unweathered samples of American upland cottons and probably among cottons generally are commonly in large measure reflections of differences in surface area per unit fiber weight. However, surface area per unit fiber weight is, in turn, frequently genetically linked or otherwise correlated with other fiber propertes, as, for example, with fiber length and strength in comparisons among cotton varieties, or with wall thickness and dyeing properties in comparisons among fiber samples of the same variety from different locations of growth. The conclusion that differences in fiber behavior or properties are related in a direct cause-and-effect manner to differences in percent wax should be approached with caution, therefore, lest the action attributed to the wax be in fact more correctly attributable to some other fiber property with which percent wax is associated in the fibers under consideration. Thus, to mention but one example, the observation of Ahmad and Sen [2] that the feel of cotton is correlated with percent wax has been amended by the further comment of Nanjundayya [19] that, although dewaxed cotton is in fact harsher to the feel than the original fiber, all cottons have approximately the same thickness of wax per unit surface area, and that, therefore, differences in feel between different un-dewaxed cottons may be related more logically in a causal fashion to the physical properties of the fiber than to its wax content.

Certain workers have been interested in the question of the degree to which measurements obtained with the arealometer represent a true index of fiber surface per unit weight as distinct from fiber shape and other possible interfering factors in the method. The high degree of correlation found in the present work between percent wax and surface area per unit fiber weight would seem to indicate that, at least in the present tests, such interfering factors were not sufficiently important to prevent the readings from yielding reasonably satisfactory relative measures of fiber surface per unit weight.*

Acknowledgment

The authors wish to acknowledge with thanks the cooperation of members of the University of Tennessee Fiber Research Laboratory in carrying out the determinations of surface area per unit fiber weight and of the following U.S.D.A. field workers who provided or assisted in the collection of the samples: J. B. Dick, D. C. Harrell, G. J. Harrison, J. G. Jenkins, W. H. Jenkins, A. R. Leding, and W. H. Tilley.

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* For additional discussion of wax content of cotton fibers see "Letters to the Editor," page 297.—*Editor.*

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(*Manuscript received January 8, 1950.*)

Letters to the Editor

Wax Content of Cotton Fibers

BUREAU OF PLANT INDUSTRY, SOILS, AND AGRICULTURAL ENGINEERING, AGRICULTURAL RESEARCH ADMINISTRATION, U. S. DEPARTMENT OF AGRICULTURE
Beltsville, Maryland
January 6, 1950

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

Subsequent to the preparation of our manuscript "Wax Content as Related to Surface Area of Cotton Fibers," which appears on page 288 in this issue, there appeared a Letter to the Editor from Dr. A. N. J. Heyn under the title "Relationship of Wax Content to Maturity of Cotton." *

Heyn reported that the wax content of cotton fiber obtained from bolls collected at intervals during the period from 25 to 50 days after flowering decreases regularly as the age of the fiber increases. The thought is expressed that this progressive decrease in percentage of wax reflects the fact that the wax is laid down entirely in the primary wall and that its percentage in the fiber varies inversely with and as a result of the deposition of the nonwaxy or cellulosic secondary wall. This interpretation is in accord with the results brought forward in the present paper and with the findings of earlier workers [2, 3]. The present authors feel, however, that in his letter Dr. Heyn has inadvertently used the terms "perimeter" and "surface area" in a loosely interchangeable manner and the term "cell wall material" in a geometrically ambiguous sense and that, although the general trend of his thought is apparent, his terminology may have a tendency to obscure certain important considerations, particularly the relationship between percent wax and the results obtained with the arealometer.

"Surface area" is a two-dimensional quantity and the term has been used for several years in expressing the results of arealometer determinations, which have been recorded in the dimensions of $cm.^2/mg.$ [4, 5]. "Perimeter," on the other hand, is a linear quantity, which may be expressed in microns or some other unit of linear measure. For a given sample of cotton fiber, the surface area per unit weight is equal to the product of the perimeter of the fiber and its length per unit weight. Perimeter may be calculated therefore from surface area per unit weight and weight per unit length; such calculations have been

* TEXTILE RESEARCH JOURNAL **19**, 711–14 (Nov. 1949).

made using data obtained by the use of an arealometer and by the usual mechanical weight-per-inch methods, and it has been brought out that genetic differences in perimeter exist in cottons [1]. Perimeter and surface area per unit weight are, therefore, not equivalent terms and are not related to one another by any fixed proportionality constant.

If one follows Dr. Heyn when he states (p. 711), "The assumption was made that the amount of wax in the primary wall is proportional to the wall surface," it is clear that his "average maturity" is for all practical purposes an attempt to measure by an indirect method the same quantity as is claimed to be measured by the arealometer—namely, surface area per unit fiber weight—and that his "average maturity ratio" is formally equivalent to a ratio which could be obtained by simply dividing a figure for surface area per unit weight from an arealometer determination on a standard sample by the same for an unknown sample to be tested. Granted that in actual practice these relationships between percent wax and surface area per unit weight by arealometer determinations do not hold with rigid or even, in some cases (weathered fiber), approximate exactness, the formal relationships need not be obscured by ambiguity in terminology.

In a practical sense percent-wax determinations as indicators of fiber maturity suffer not only from the disadvantage mentioned by Dr. Heyn—namely, length of time required per determination—but also from the factor of expense of solvents, possible fire hazard, space required, and the limitation that only unweathered samples may be used if one is to have any confidence in the results. There is also evidence, as herein presented, to indicate that wax per unit surface area on even unweathered fibers is not entirely constant. By contrast with the wax method, the arealometer method is rapid, requires essentially no expenditure for materials (in addition to the original cost of the instrument), may be used on a very small sample, and presents no hazards to the operator.

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PAUL B. MARSH
HENRY D. BARKER
THOMAS KERR
MARY L. BUTLER

The Sub-Cuticle Membrane—A Recently Discovered Morphological Component of the Wool Fiber

RESEARCH DEPARTMENT, WOLSEY LTD.
Leicester, England
December 28, 1949

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

Reumuth [9] as a result of microscopic examinations of damaged wool fibers concluded that there was a sheath-like layer between the scales and the cortical cells which represent the main body of the fiber. Lehmann [4] confirmed this claim; he isolated a membrane, which he called the "sub-cutis" (representing about 5% of the total wool substance), by dissolving the remainder of the fiber in a formaldehyde solution at 140°C. Elod and Zahn [3, 10] obtained the same material by different although also very drastic chemical processes. Because these methods of preparation were so severe and gave results which were far from unambiguous, the existence of this membrane was not generally accepted. Lindberg, Philip, and Gralén [7] demonstrated electronmicroscopically the existence of a non-protein, chemically very resistant sheath, approximately 100 Å. thick (*i.e.*, approximately 0.1% of the wool as a whole), which was situated over the scales and which

they called the "epicuticle." Following on this work these authors [8] questioned the existence of a sub-cuticle membrane, suggesting that it consisted of the epicuticle thickened by the localized adhesion of underlying scale substance. They claimed that the membrane as isolated by the methods of both Lehmann and Elöd and Zahn looked like the external surface of the fiber, still showed scale edges, and was of non-uniform thickness.

We have discovered an unobjectionable method of separating a membrane which may be identical with the "sub-cutis" by treating wool with peracetic acid, which was shown [2] to confine its attack almost exclusively to the oxidation of the disulfide bond to sulfonic acid groups. After more than 90% of the cystine has been oxidized with this reagent (*e.g.*, by treatment in a 1.6% aqueous solution of peracetic acid for 25 hrs. at room temperature), the wool becomes freely soluble in dilute ammonia except for a resistant fraction which represents between 7% and 10% of the wool substance and which is in the form of a membrane (see Figure 1). The soluble fraction is precipitated as a white powder on acidifying the ammonia extract. The insoluble residue can be repeatedly re-treated with peracetc acid followed by ammonia extraction without dissolving any of it, and there can be no doubt that it represents a distinct fraction of greater chemical resistance than the remainder of the wool.

The membrane is continuous, runs along the whole length of the fiber, and when its two ends are closed with micro-manipulators osmotic swelling effects can be seen under the microscope on the addition of electrolyte solutions. As is evident from Figure 1, there is no sign of scale edges or, in fact, of any lateral markings. The membrane also is quite resistant to chlorine solutions of any pH, whereas if it consisted of scales held together by non-protein epicuticle it would collapse, since by this treatment the latter would be detached from the wool [5].

By observing microscopically the dissolution of the oxidized fiber in ammonia, the scales can be seen to disappear rapidly, leaving no lateral markings of any kind. After this, the solution of the wool substance is slow, probably because it has to diffuse out through the open ends of the resistant tube. It is not possible to ascertain the position of the membrane in the fiber with any certainty, for, as Lindberg *et al.* [6] have shown, the so-called scales are, in fact, of a highly complex histological structure. Reumuth's

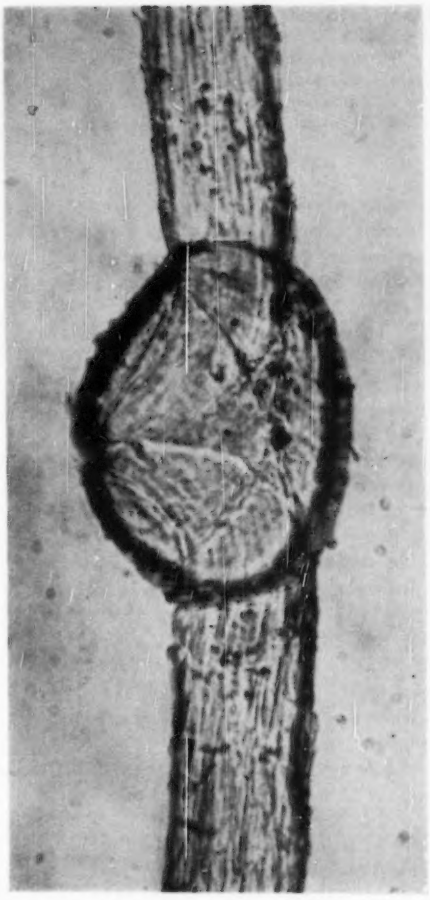

FIG. 1 (a). *The fiber after oxidation with peracetic acid. There was no weight loss.*

[9] view that it is located between the scales and the cortical cells is compatible with our findings.

Preliminary experiments on the constitution of the membrane by paper chromatography show it to be a protein—the possible small amount of contamination due to the epicuticle would not be detected. It contains no cystine, which has been completely oxidized during the preparation, but some cysteic acid (corresponding to about 3% cystine) has been found, although most of it was probably dissolved in the ammonia during extraction. Otherwise the membrane contains all the amino acids present in the wool fiber

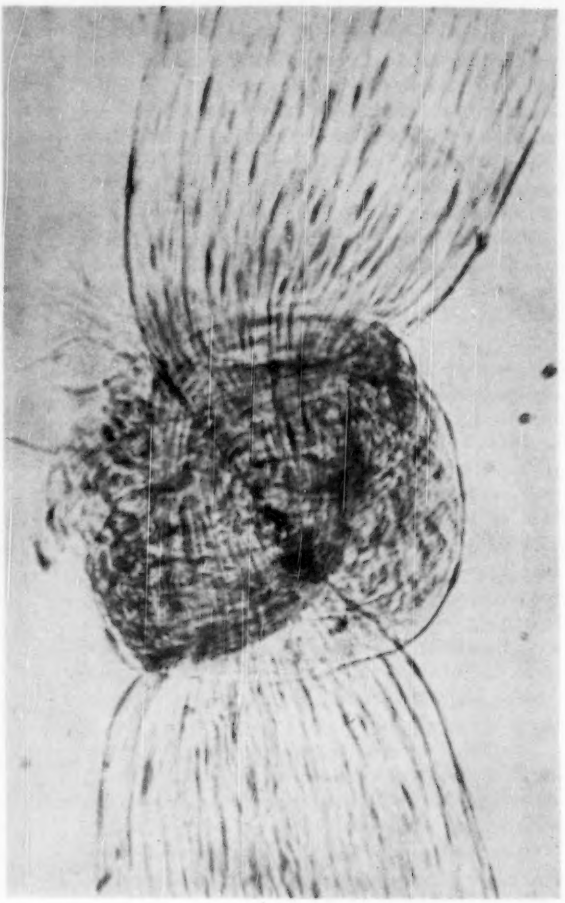

FIG. 1 (b). *The insoluble remainder after extracting the fiber shown in* (a) *for 14 hours in N/10 ammonia. No change occurred in a further treatment with peracetic acid followed by extraction with ammonia. The fiber was knotted so as to show the continuity of the membrane.*

as a whole, and no others could be detected. They are not, however, present in the same proportions (*e.g.*, the content of serine is higher but that of glycine and alanine is lower).

The resistance of the membrane as isolated (*i.e.*, after oxidation) to alkali is similar to that of untreated wool and it dissolves slowly in *2N* sodium hydroxide and sodium sulfide.

The reason for the increased chemical resistance of the membrane, which will have to be considered as distinct protein, is of the greatest interest and cannot be due to powerful hydrogen bonding, since it is insoluble in strong solutions of lithium bromide [1]. It seems likely that the chains are held together by a covalent cross-link (in addition to the disulfide bond), which is perhaps an ether linkage and which is not present in the remainder of the wool.

The sub-cuticle membrane must exert an important influence on the physico-chemical behavior of wool, and we believe that it can act as a selective membrane which, in alkaline solution, for example, will have a negative charge and retard the diffusion of anions into the fiber.

Thanks are due to Mr. M. Fox for carrying out the paper chromatography and to the directors of Wolsey Ltd. for permission to publish this letter.

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P. ALEXANDER
C. EARLAND

INDUSTRIAL SECTION

Effect of Crimp on Fiber Behavior

Part I: The Determination of Fiber Irregularities and the Concepts of the Single-Fiber Bulking Capacity*

L. W. Rainard† and Dorothea Abbott‡

Alexander Smith & Sons Carpet Company, Yonkers, New York

Abstract

An objective method for determining fiber irregularities is suggested wherein it is recognized that there are many instances of crimpiness which cannot be defined in terms of frequency and amplitude measurements. An attempt was made to arrive at a simple correlation between roving diameters and single-fiber properties based on a geometric analysis of a roving. An empirical expression is presented which to a fair degree allows the prediction of equilibrium roving diameters from measurement of the properties of single fibers. A laboratory technique for studying the bulking capacity of wools is presented which is useful possibly as an objective technique for evaluating what potentially is of greater technical significance than crimp—namely, the bulking tendencies of irregularly shaped fibers.

Introduction

Regularly crimped fibers can be characterized quite easily; however, many fibers have very irregular crimp which cannot be defined by simple frequency-amplitude relationships. As has been shown, crimped nylon and rayon staples have, for certain uses, more desirable processing and product properties than uncrimped staples. Moreover, natural fibers which possess crimp differ from each other in the amount of crimp. Therefore it appears to be important to determine the degree of crimpiness and its relationship to the observed differences in process and product.

Up to this point, in the conventional methods of approaching the problem the crimp has generally been considered to be a regular occurring sine wave which can be defined by a frequency and amplitude determination. However, individual fibers do not always have a regular crimp as to frequency, shape, or amplitude. For example, the India (*e.g.*, Joria) wools (Figure 1) have such an irregular crimp that it is really most difficult to say where one crimp ends and the next one starts. This problem exists also to some extent with rayons that are crimped by relaxation. The measurement of frequency and amplitude generally is a subjective procedure requiring a choice on the part of the operator and has no definite basis. Hence, a more completely objective technique would be desirable.

Crimp in a fiber, whether it be regular or irregular, can be defined as the degree of deviation from linearity which the fiber possesses. However, this deviation from linearity is important mainly in the over-all fiber direction, since it is this deviation or apparent bulkiness of the fiber which may be of

* The material in this report was presented at the Spring Meeting of The Fiber Society, Fontana Village, N. C., April 27, 1950. Part II follows on page 308 of this issue.

† Chief, Chemical & Products Research Section, Research & Development Division.

‡ Assistant Chemist.

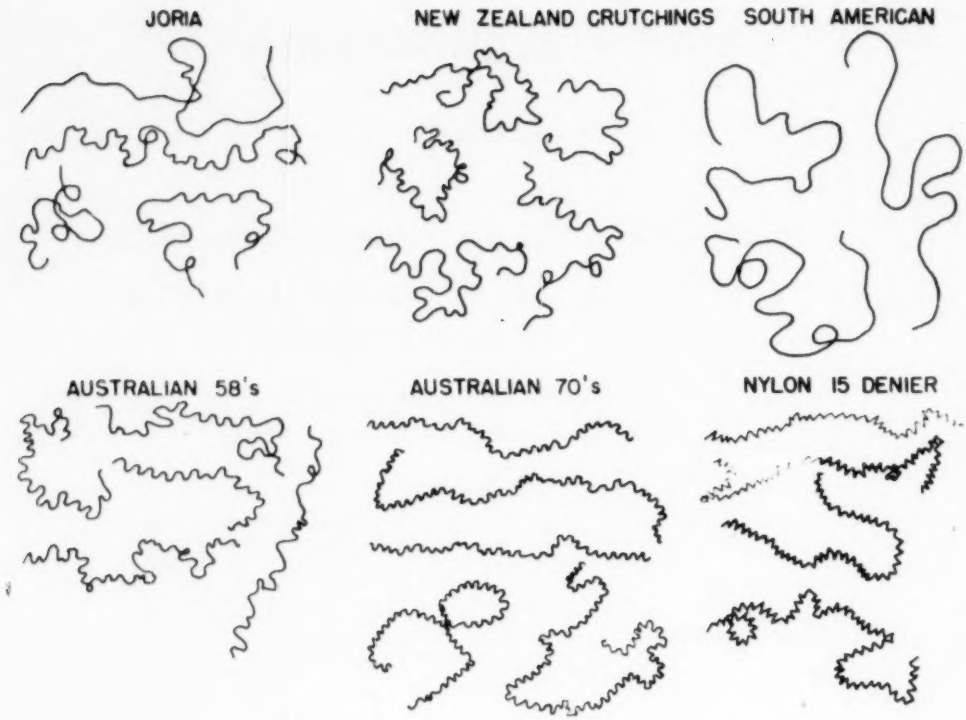

FIG. 1. *Variation in crimp type.*

value in altering the characteristics of rovings and yarn.

If one considers the general conditions under which fibers are used, it will be seen that the evaluation of shape characteristics of unrestricted fibers can easily be meaningless. For example, if we consider fibers with one or two large bends, these bends are not of general importance since they can easily be straightened out in processing. However, a fiber with a large number of bends, such as the Australian 70's, is liable to have a shape effect which will persist throughout processing with only minor modifications. Therefore, large bends seem useless, whereas fine crimps seem useful. One problem, then, is simply to determine when the size of crimp becomes important. Before answering this question, conditions of use must be defined and analyzed for their possible relationship to the properties of single fibers.

To this end one major restriction is imposed—namely, that under the conditions of measurement the fiber must lie roughly in a volume of approximately the size of a roving or yarn. This condition merely sets up an artificial restriction so that the fiber is generally lined up to minimize the large bends. This is accomplished experimentally by placing the fiber in a long slot the width of which is roughly the width of a standard roving, which will be described later (Figure 2). This slot represents the maximum variation in direction which a fiber can possess in the roving of that particular size. For the purposes of this study these variations in direction are further restricted to one plane. Now, if we superimpose on the fiber a system of parallel lines whose spacing is generally smaller than the smallest crimp, we can get some idea as to the distribution of the fiber mass over this specified area, observing the average number of intersections which the fiber makes with the lines in this system of parallels. Then, by arbitrary definition, the bulking capacity (B) of a single fiber is defined in equation (1) (see

Appendix) as the average number of intersections of the fiber with the grid system per unit of length. Assuming that this quantity B is directly related to the effective fiber volume, V_F, as shown in equation (2), we can define for any given roving the volume per unit length, V_R, as the average effective volume of the single fibers, V_F, times the total number of fibers in the cross section, N_F (equation (3)). Considering the roving as a cylinder, then, the diameter, D_R, is given as indicated in equation (4). Equation (5) gives the number of fibers in cross section per unit length of roving in terms of the weight of the sample and the length of the roving. Substituting in equation (4), the expression for the diameter of the roving is obtained in terms of weight of the sample, effective fiber volume, or the bulking capacity of single fibers, and fiber diameter, equation (6). Since equation (6) contains constants which must be experimentally determined and other geometric factors which cannot be easily included in this equation but which would represent a constant source of error, it is simpler merely to compare the ratios of the roving diameters of two fibers (A and B) as seen in equation (7), where the constants of equation (6) cancel out. A simple expression is obtained involving the effective fiber volume or the bulking capacity of the fiber and its corresponding fiber diameter.

Single-Fiber Bulking-Capacity Measurement

For India, New Zealand, and South American wools, about 25 g. of each of the wools was hand-carded. Then 15 small bunches of the wool were picked at random from the large bulk of carded wool. These bunches were combined and hand-carded. The wool was then drawn into a roving with the hand rub aprons. In order to extract a sample from the roving, a threaded needle was inserted into the roving and a section was tied off and removed. All fibers not held by the thread were gently combed out. Fibers looped through the thread were not removed. All fibers within the bundle were measured.

Nylon, Australian 58's, and Australian 70's fibers seemed to be so similar in crimp that the above technique was not followed. For each wool a small pinch of fibers was removed from a thoroughly carded sample, and every fiber in the pinch was measured.

For measurement, each fiber was mounted in a trough. The trough was constructed from two strips of wood placed parallel 2.0 mm. apart on a glass plate. The strips of wood were 4.5 mm. high and 100 mm. long. Thus, the trough was 4.5 mm. deep and 2 mm. wide. One end of the fiber was fastened to the glass bottom of the trough with a small piece of scotch tape. The other end of the fiber was stretched out straight inside the box and held there while a strip of highly polished Plexiglas (4.0 mm. high, 2.0 mm. wide, and 100 mm. long) was placed along the open top of the trough to form a cover. Then the fiber was released and allowed to relax within the trough. The strip of plastic was then pressed down into the trough, thereby flattening the fiber into a plane. The glass plate holding the trough was then placed in a photographic enlarger. The fiber image at a known magnification was projected onto graph paper, with the lines of the paper in one direction placed parallel to the side edges of the trough. Every intersection of the fiber and the grid lines was marked on the paper. The length of the fiber in grid units and the number of grid lines intersected by the fiber were also noted. Some of the longer fibers of the Australian wools and nylon were cut to a convenient length.

Roving-Diameter Measurement

In order to relate this measurement of single-fiber bulkiness to roving diameters, some uniform and reproducible technique for making roving was needed.

Each sample was taken from the carded wool used for bulk density measurements. The wool was

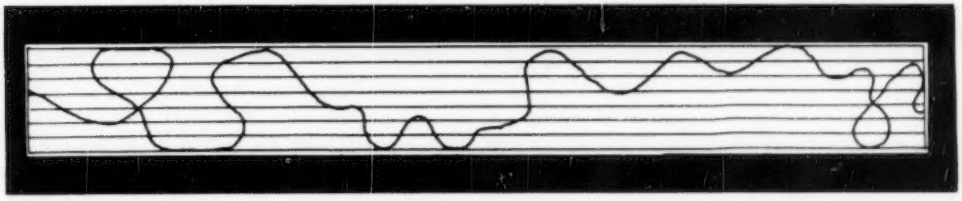

FIG. 2. *Fiber mounted in slot and superimposed on grid system.*

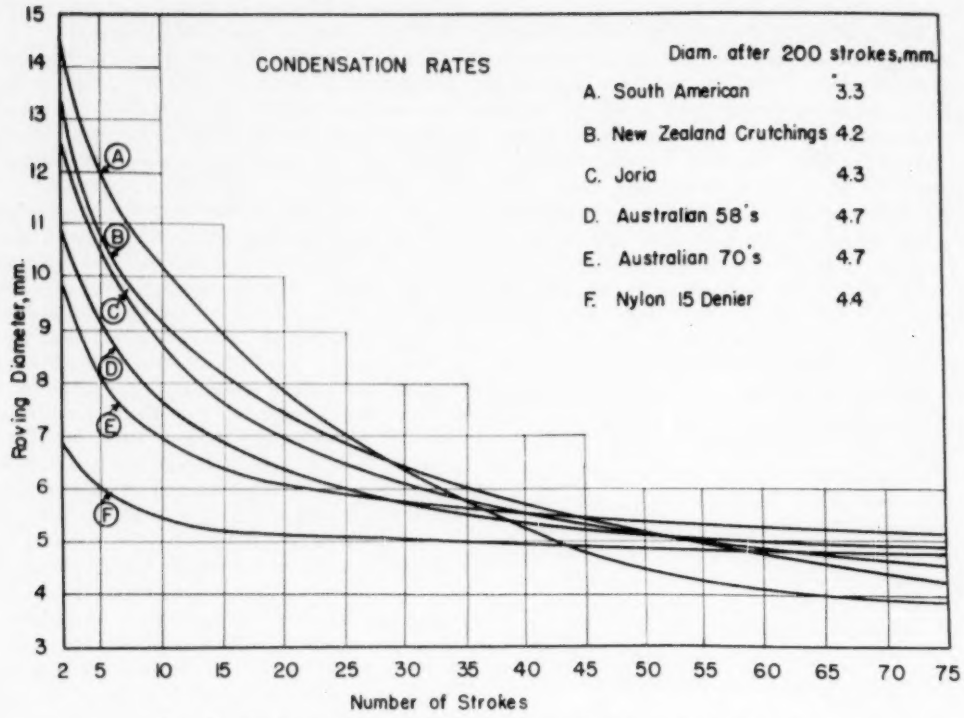

FIG. 3. *Condensation rates for various fibers using short rub-apron stroke.*

combed in order to get all the fibers to lie parallel and five 60-mg. bundles were extracted. The wool was conditioned for at least 12 hrs. at 72°F and 52% R.H.

A set of hand rub aprons were built of wood and covered with leather from a mill rub apron.

The dimensions of the boards, not including the leather (0.3 cm. thick), were: upper apron, 26.5 × 13.7 × 1.8 cm.; lower apron, 26.5 × 16.0 × 1.8 cm. A handle was attached to the upper apron. Its weight was 555 g.

The lower rub apron was mounted on a large board and pins were placed along the two longer sides of the apron in order to give each stroke a definite width.

Each of the five 60-mg. bundles of fibers was placed on the lower rub apron so as to cover an area 8.5 cm. long and 2.5 cm. in width. The upper apron was rubbed across the lower, using no downward pressure and only enough sideward pressure to move the apron. After a definite number of strokes, the diameters of the roving were measured. Diameter measurements were made in three places along the roving (at approximately the lines dividing the roving into quarters). The roving was then carefully rolled through a 90° angle and 3 measurements of diameter were made. Measurements were made in two directions in order to compensate for the tendency of some rovings to flatten out. Each plotted point represents 30 readings—*i.e.*, 6 readings for each of 5 bundles.

Two separate sets of condensation curves were obtained. One set was obtained using 30-mg. bundles and a long stroke (apron moves a total distance of 36 cm. per stroke) and taking readings at 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 20, 30, and 50 strokes. The second set of curves was obtained using 60-mg. bundles and a short stroke (6 cm.), with readings taken at 2, 4, 6, 8, 10, 15, 20, 25, 30, 35, 40, 50, 75, 100,

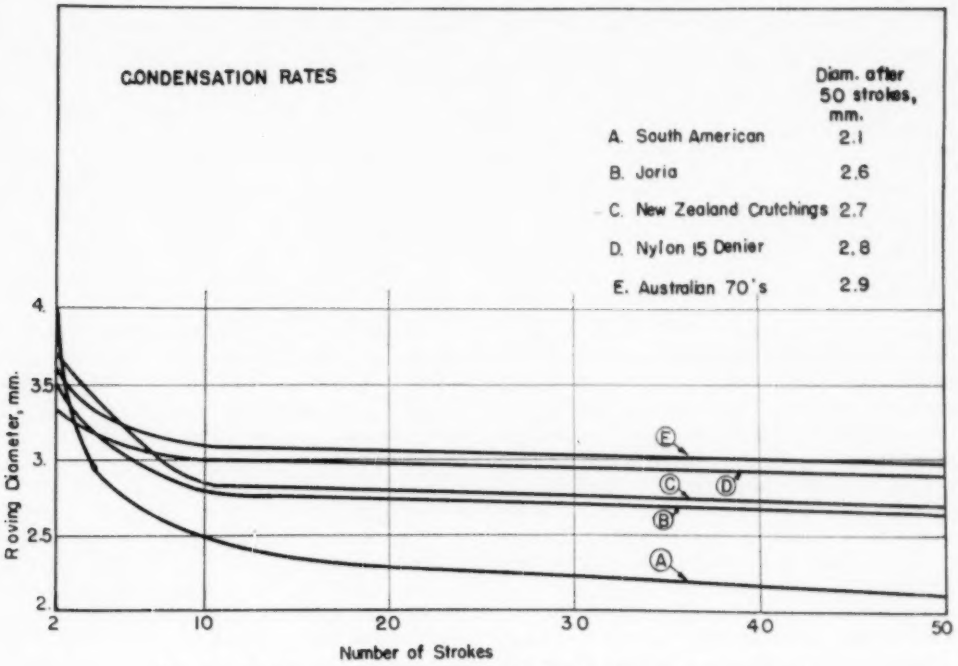

FIG. 4. *Condensation rates for various fibers using long rub-apron stroke.*

150, and 200 strokes. These curves are plotted in Figures 3 and 4.

This technique indicates that there is a real equilibrium roving diameter which is effective independent of the total amount of work done on its system. It also indicates that the rate at which equilibrium is reached is also related to equilibrium roving diameter and general crimpiness. Table I lists the equilibrium roving diameters, the fiber diameters, and the bulking capacities for a group of fibers ranging from very coarse India wools to the fine Australian wools and including 15-den. nylon.

TABLE I. EQUILIBRIUM ROVING DIAMETERS, FIBER DIAMETERS, AND BULKING CAPACITIES FOR VARIOUS FIBERS

Fiber	B	D_R (mm.)	D_F (μ)
South American	.149	3.25	34.8
Joria	.264	4.30	33.5
New Zealand crutchings	.273	4.15	39.2
Nylon, 15 den.	.286	4.39	36.3
Australian 70's	.225	4.72	20.1
Australian 58's	.319	4.67	24.2

The correspondence between the ratio of the actual roving diameters and the calculated ratio, shown in Table II, is fair in the class of wools which have a large diameter and, seemingly, it does not apply to the Australian types of wool. An empirical equation (equation (8)) has been worked out which gives a satisfactory correspondence over all ranges of fiber diameter used in these studies. This empirical equation essentially removes the parameter of fiber diameter in equation (7).

Examination of the data in Tables II and III will show that the very fine wools do not have as high a bulking capacity as calculated. This presumably is due to the fact that as the fiber diameter decreases, the total number of fibers per unit weight increases, and a simple multiplication of the single-fiber bulking capacity by the number of fibers should give the roving diameters. This discrepancy is probably due to the fact that our equations do not take into con-

TABLE II. CORRESPONDENCE BETWEEN RATIOS OF ACTUAL ROVING DIAMETERS, $\frac{D_{RA}}{D_{RB}}$, AND RATIOS OF CALCULATED ROVING DIAMETERS, $\frac{D_{FB}}{D_{FA}}\left(\frac{B_A}{B_B}\right)^{\frac{1}{4}}$ (FIBER B IS SOUTH AMERICAN AND IS CONSTANT)

Ratio	$\frac{B_A}{B_B}$	$\frac{D_{RA}}{D_{RB}}$	$\frac{D_{FB}}{D_{FA}}\left(\frac{B_A}{B_B}\right)^{\frac{1}{4}}$
Joria : S.A	1.78	1.32	1.38
N.Z.C. : S.A.	1.84	1.28	1.21
Nylon : S.A.	1.92	1.35	1.34
70's : S.A.	1.52	1.45	2.13
58's : S.A.	2.14	1.43	2.10

TABLE III. CORRESPONDENCE BETWEEN RATIOS OF OBSERVED ROVING DIAMETERS AND RATIOS CALCULATED FROM AN EMPIRICAL EQUATION (EQUATION (8))

$$\sqrt{\frac{B_A}{B_B}}\frac{}{D_{FA}} + K = \frac{D_{RA}}{D_{RB}}$$

Ratio	Calculated	Observed
Joria : S.A.	1.32	1.32
N.Z.C. : S.A.	1.27	1.28
Nylon : S.A.	1.31	1.35
70's : S.A.	1.53	1.45
58's : S.A.	1.53	1.43

sideration the bending modulus or other forces required to bend a fiber. In the case of very fine fibers these forces would be small, so that multiplication of the single-fiber bulking capacities by the total number of fibers would be erroneous because the bulking capacity obviously must be different in the roving form from what it is for the free fiber. Whether or not this empirical equation is applicable to all cases under other conditions of test has not yet been determined.

Attempts will be made to derive expressions on a theoretical basis which use other parameters known at present to be operative in affecting bulkiness. However, for the purposes of this investigation, equation (8) gives fair correspondence between predicted and observed values (Table III).

One of the reasons why the simple geometric classification of effective fiber volume is unsatisfactory is that our method of measurement assumes that this effective volume will persist, being only partially modified during processing. Actually, it is not difficult to conceive of conditions whereby a high degree of regularity of crimp would cause closer packing simply by shifting portions of the fiber one-half wave length along the fiber direction. Another factor to be considered is the effect of the bending modulus and the ability of a single crimp to stand off its neighbors. If the bending modulus is high enough, presumably a few crimps would be sufficient to create some bulking effect. If, however, the bending modulus is extremely low, under the conditions of condensing or spinning that effect would be lost. The equations used in this discussion of crimp omit the bending modulus factor. However, in the near future an attempt will be made to include this factor.

Appendix

Formulas Used

$$\sigma = \sqrt{\frac{\Sigma\ (\text{Deviations from } \bar{X})^2}{\text{Number of readings}}}$$

$$\text{Coefficient of variation} = \frac{\sigma}{\bar{X}} \times 100$$

$$90\%\text{ confidence limits on mean} = m \pm \frac{\sigma(1.645)}{\sqrt{n-3}}$$

$$50\%\text{ confidence limits on mean} = m \pm \frac{0.6745\sigma}{\sqrt{n}}$$

Equations

(1) $B = \frac{I}{nl}$, where I = number of grid lines intersected; n = total number of intersections per fiber; l = length of fiber

(2) $V_F = KB$

(3) $V_R = V_F N_F = KBN_F$

$$(4) \qquad D_R = \sqrt{\frac{4V_R}{\pi}}$$

$$(5) \qquad W = \frac{\pi}{4} D_F^2 \rho L N_F$$

$$(6) \qquad D_R = \frac{4}{\pi D_F} \sqrt{\frac{KBW}{\rho L}}$$

$$(7) \qquad \frac{D_{RA}}{D_{RB}} = \frac{\frac{\sqrt{B_A}}{D_{FA}}}{\frac{\sqrt{B_B}}{D_{FB}}} = \sqrt{\frac{B_A}{B_B}} \times \frac{D_{FB}}{D_{FA}}$$

$$(8) \qquad \frac{D_{RA}}{D_{RB}} = \frac{\sqrt{\frac{B_A}{B_B}}}{D_{FA}} + K$$

Statistical Analysis — Bulk Density Measurements

Fiber	$\bar{X}$	σ	Coeff. of variation	Limits statistical probability = .90	Limits statistical probability = .50
India	.112	.059	52.7%	±.015	±.006
S. A.	.063	.034	54.0%	±.008	±.003
N. Z.	.116	.039	34.1%	±.011	±.004
Nylon	.121	.033	27.8%	±.010	±.004
Australian 70's	.095	.019	20.0%	±.007	±.002
Australian 58's	.135	.037	27.4%	±.010	±.004
Condensation Rates					
S. A.					
6 strokes	11.5	4.22	36.7%	±1.37	±.518
200 strokes	3.25	.45	13.8%	± .143	±.054
India					
6 strokes	9.94	2.39	24.1%	± .760	±.281
200 strokes	4.30	.58	13.6%	± .185	±.071
N. Z.					
6 strokes	10.30	2.20	21.4%	± .696	±.270
200 strokes	4.15	.34	14.3%	± .109	±.042
Nylon					
6 strokes	5.76	.96	16.6%	± .304	±.120
200 strokes	4.39	.65	14.7%	± .206	±.080
Australian 70's					
6 strokes	7.54	1.48	19.7%	± .469	±.184
200 strokes	4.72	.75	16.0%	± .238	±.092
Australian 58's					
6 strokes	8.62	1.54	17.9%	± .488	±.189
200 strokes	4.67	.79	15.9%	± .248	±.097

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Effect of Crimp on Fiber Behavior

Part II: Addition of Crimp to Wool Fibers and Its Effect on Fiber Properties*

J. L. Barach† and L. W. Rainard‡

Alexander Smith & Sons Carpet Company, Yonkers, New York

Abstract

The importance of crimp in natural wools is discussed. Wear and coverage of crimped *vs.* uncrimped materials are demonstrated. A method of crimping wool artificially is shown and production equipment is discussed schematically. Theoretical expressions are developed for stress-concentration effects and are compared with experimental results. A theory for the mechanics of the crimping operation is advanced.

THE OPENING STATEMENT in Harold De Witt Smith's monumental Marburg Lecture essentially describes the motivation for the work which is reported here—"There is no such thing as a natural textile fiber! There are only natural fibers which have been selected by mankind for use in textiles." In the section of Smith's Marburg Lecture which deals with the influence of fiber shape on fiber behavior, there is a rather extensive analysis of fiber shape in terms of variations in cross section. However, apart from minor mention, the element of fiber shape taken lengthwise, as opposed to the cross section, has not been studied in general, and no attempt has been made to evaluate quantitatively the effect of variation of fiber shape in the longitudinal direction on the performance of a fiber.

Using the engineering approach to the utilization of natural fibers, we have developed a method which offers potentials for overcoming the natural deficiencies of certain fiber types where crimp is a factor limiting its usage.

There is some general knowledge that crimp contributes something and that it is a necessary property of fibers. Certain synthetic fiber producers consciously add crimp in order to increase the bulkiness of top or to change the hand of fabric, but, as far as we have been able to determine, this has been done without accurate knowledge as to the effects of such gross deformation of the fiber. In the woolen field, historically, there have been available for a considerable length of time fibers which are highly crimped and others which are long and silky, such as the mohair and the South American cross-bred wools. Some of these are illustrated in Figure 1 of Part I (page 302). It has been known for a long time that the cross-bred wools were definitely not so desirable for certain uses as the Merino types. Actually, there have been attempts to correlate fiber fineness with the degree of crimp present in the fiber, but these have failed except within classes of a single wool type [1].

There is a history of the use for various purposes of over two hundred carpet wools—the specific use depending, of course, upon availability of these wools. The normal blend for carpet use consists of five or six different types of wool. Blending is necessary in order to obtain coverage, to provide abrasion-resistance or to increase wear life, to make economical spinning feasible, to obtain a desired color, and for other reasons.

In this paper we shall attempt to correlate variation and degree of crimp with resultant changes in processing and product characteristics.

The carpet industry uses considerable amounts of a long, straight fiber which is imported primarily from South America. This wool has notoriously poor spinning properties when used unblended with other fibers, giving a yarn which is extremely thin for its weight, with very poor coverage in pile fabrics

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† Chief, Physical Research Section, Research & Development Division.

‡ Chief, Chemical & Products Research Section, Research & Development Division.

and with surprisingly poor wear for what seems to be a chemically sound wool. Since this fiber has been available in large quantities at relatively low price, its utilization has constituted a problem. However, it is one thing to indicate that crimp in a fiber will contribute to the bulkiness or fullness of the yarn and the fabric made from it, and another to obtain crimped fibers from materials which naturally occur in a long, silky, straight form. To this end we have devised laboratory-wise and, at present, large-scale production equipment for the crimping of naturally resilient fibers.

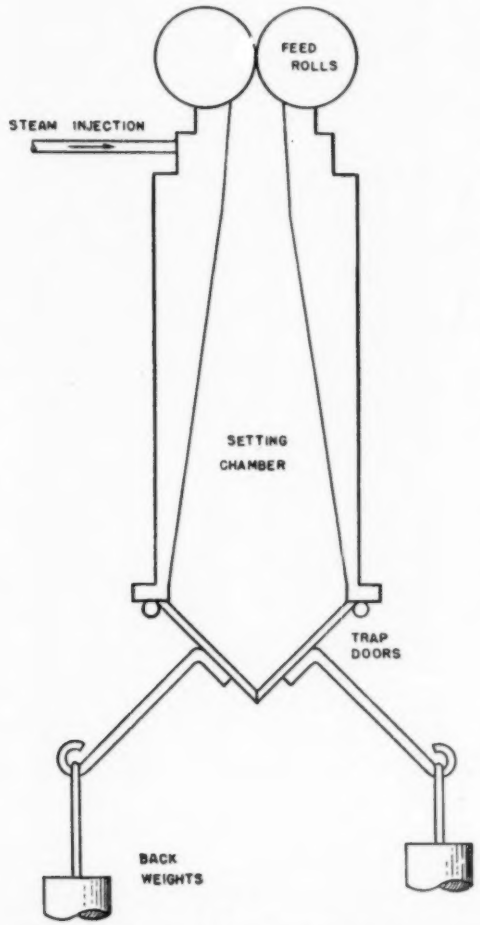

FIG. 1. *Schematic diagram of crimping apparatus.*

This apparatus is shown schematically in Figure 1. The feed rolls of a stuffer crimper force fibers in slubbing form into a chamber with a restricting outlet, in this case a pair of symmetrical hinged doors which are weight-loaded. The restriction of fiber flow causes the chamber to become filled with densely packed fiber. As new fiber is continuously fed in, the new fiber must collapse against the fiber mass already in the chamber, causing an alternating folding of the fibers, which results in an angular crimp. Our apparatus operates in a mechanically similar way to the conventional stuffer box crimper, with two important exceptions: first, the equipment is greatly enlarged in order to handle production at rates greater than 300 lbs. per hr., and, second, means are provided whereby naturally resilient fibers such as wool, mohair, and silk can be set in the crimped state. This is accomplished by a series of openings in the upper portion of the stuffer chamber wherein setting agents, such as high-temperature steam or solutions of reducing agents, can be introduced while the fiber is constrained in the crimped form. A balance of forces is maintained in the crimper by trap doors in the bottom which keep the apparatus in continuous operation. The remainder of the chamber is greatly enlarged to allow for contact periods of anywhere from 30 sec. to 3 min. That this length of time is sufficient for setting this crimp may be seen by examination of Figure 2. Here are shown roving diameters after different periods of boiling for typical materials.

The development of this test method is discussed in detail in Part I of this paper (page 301). In most cases a 1-hr. boil will be as much or more than a fiber will ever be subjected to during processing and use. As Figure 2 indicates, we have here a reasonable increase in roving diameter over untreated wool for all cases.

When the fibers emerge from this modified stuffer crimper they are impressed with a permanent angular crimp. Of necessity, we have chosen a particular configuration for our experiments and usage. This configuration has been dictated to a large extent by mechanical configuration of the apparatus, but it is possible to produce a wide range of values in accordance with the fundamental properties of the materials which are put into the apparatus and the pressures, lengths of time, amounts, and characteristics of the materials used. Figure 3 is a photograph of crimped and uncrimped fibers rubbed into rovings; these

crimped rovings represent typical artificially crimped materials. These rubbed rovings afford a means of measuring the condensing ability of the fibers after crimp has been added artificially. That crimp is related to the covering power of the yarn can be seen by comparing the rubbed bundles with carpets made from similar materials and illustrated in Figure 4. The most striking difference to be observed is the

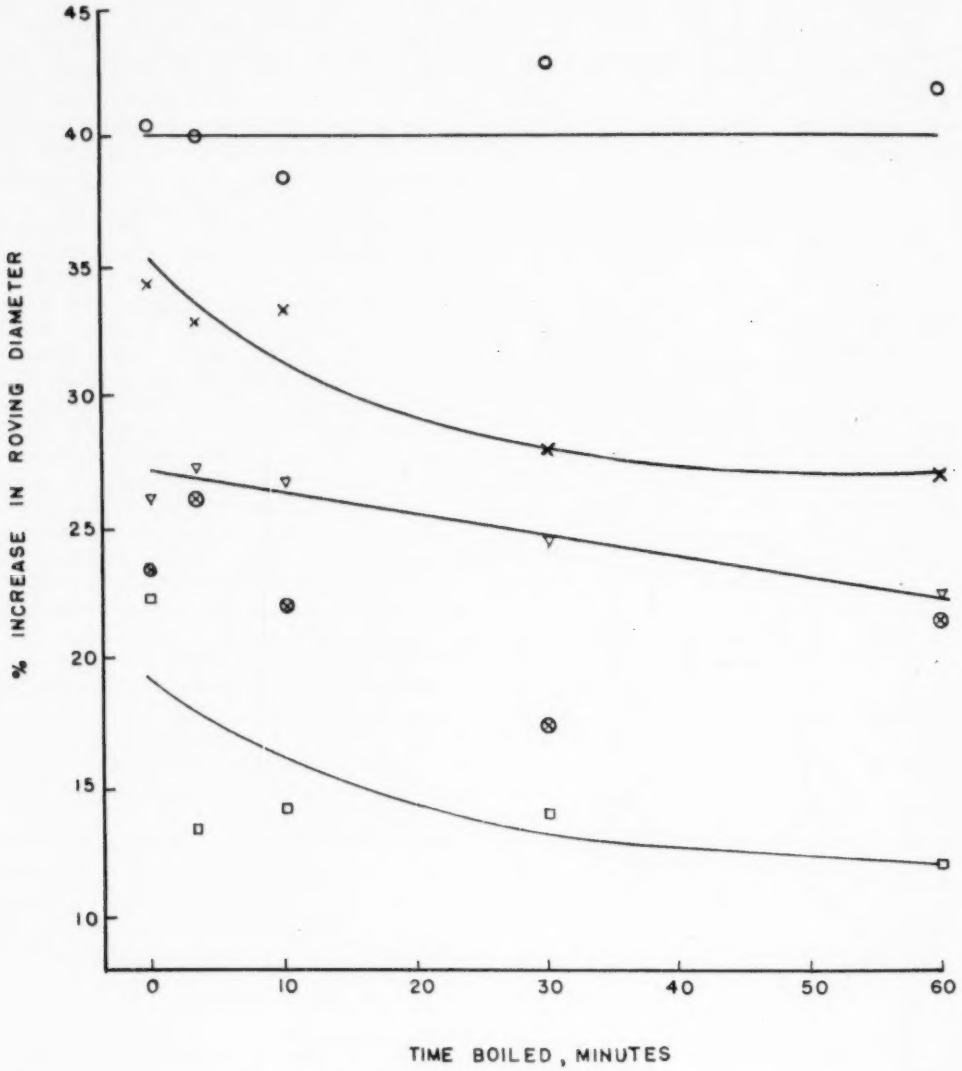

FIG. 2. *Effect of boiling on roving diameter increase (over uncrimped wools). Fiber A* ○-○*—High crimp, high permanence. Fiber B* ×-×*—High crimp, low permanence. Fiber C* ▽-▽*—Low crimp, high permanence. Fiber D* ⊗-⊗*—Low crimp, low permanence. Fiber E* □-□*—Low crimp, low permanence.*

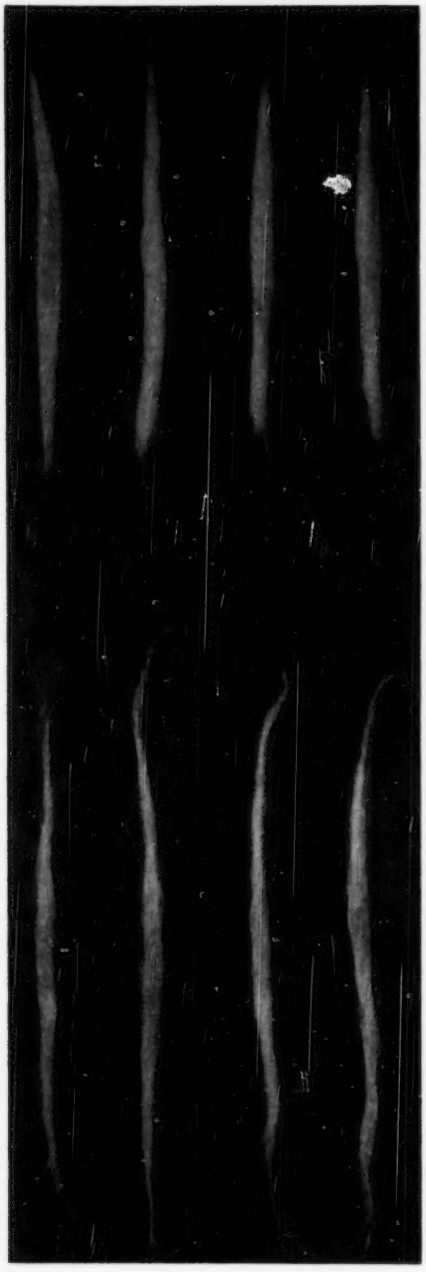

FIG. 3. *Rubbed rovings from crimped (right) and uncrimped (left) fibers.*

change in diameter of the rubbed bundles. In other words, for equal fullness of yarn or fabric it is possible to reduce the weight of the wool to be used. It should also be noted in the photograph (Figure 3) that the crimped roving has a smoother, evener, less hairy appearance than that made from uncrimped wool.

There are certain concepts used in the study of strength of materials which may be applied as an aid in the interpretation of the crimping operation. If one considers a column which is long in relation to its diameter and if one also assumes as valid the concept of slenderness ratio as it is used in column theory, then a series of equations can be developed which show how the modulus of stiffness, the fiber moment of inertia, and the back weight pressure all influence the type, size, and shape of the crimp obtained.

If we assume that the fiber in the crimper is being bent just as is a column which is fixed at both ends

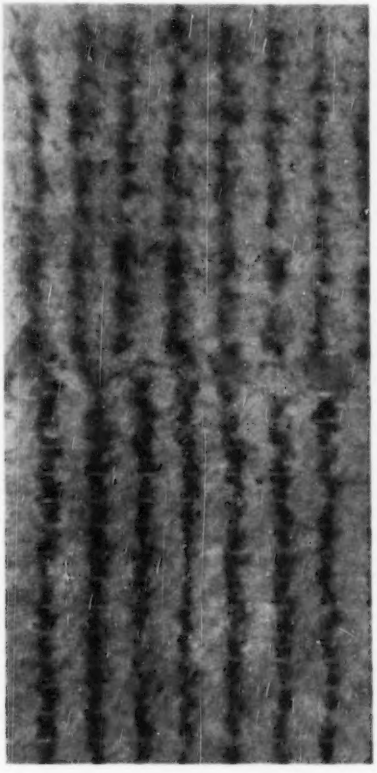

FIG. 4. *Coverage power of uncrimped (left) and crimped (right) wools.*

TABLE I. EXPERIMENTAL MEASUREMENTS FROM SELECTED SOUTH AMERICAN FIBERS

Sample No.	Diameter (μ)	Leg length (in.)	Modulus (p.s.i.)	Pressure (p.s.i.)
1	31	.0712	1.00×10^6	8.52×10^{-4}
2	31	.0575	$.366 \times 10^6$	4.64×10^{-4}
3	32	.0644	1.00×10^6	11.8×10^{-4}

TABLE II. CALCULATIONS MADE BY SUBSTITUTING VALUES IN TABLE I IN EULER'S EQUATION

$$P = 4AE\pi^2\left(\frac{r}{l}\right)^2$$

Samples	Constant	Theoretical ratio	Actual ratio
1 & 3	Radius and modulus	1.185/1	1.11/1
1 & 2	Radius	1.49/1	1.24/1
2 & 3	Radius	1/1.035	1/1.12
1 & 2	Pressure and radius	1.81/1	1.24/1

and is subjected to an axial load, Euler's formula for critical load (*i.e.*, load to collapse the column): $P = \frac{4\pi^2 EI}{l^2}$ applies. Of course, this applies accurately only as long as the load P remains within the proportional limit—that is, the linear portion of the stress-strain curve for the fiber in compression. The validity of this may also be questioned, because of the fiber interaction which takes place in the chamber. From a qualitative standpoint, however, we can calculate the effect of changing the different forces and fiber characteristics during the operation. If we rearrange the above formula and eliminate the moment of inertia, I, we can express the equation in the form $P = 4AE\pi^2\left(\frac{r}{l}\right)^2$. Since A is a constant for a particular fiber and π^2 is constant, the critical load depends upon the modulus and $\left(\frac{r}{l}\right)$—that is, the ratio of fiber radius to leg length or the straight portion between crimp. In other words, as is actually the case in the crimping operation, the compressional load upon the fiber is a constant and when $\left(\frac{r}{l}\right)$ becomes sufficiently small, as it is made to do by feeding the fiber between the crimping rolls, the fiber will collapse and a crimp will be formed. By inspection it can also be seen that the fiber diameter will determine to a large extent the crimp frequency which occurs in a particular kind of wool and that the bending modulus or stiffness E is also an important variable and one which to some extent can be controlled.

TABLE III. CARPET WEAR PROPERTIES

Wool	Wear index	Wear criterion	Wear/Unit criterion
South American	60 K	131	458
India	81.5 K	172	474
Artificially crimped S.A.			
7.3 crimps/in.	62 K	139	446
9.7 crimps/in.	59 K	123	480
11.7 crimps/in.	69.5 K	149	466

NOTE: These are selected results which we believe to be representative and which correlate with floor wear. The nomenclature used in the above table is that defined in Research Paper RP 1505, *J. Research Natl. Bur. Standards* **29**, Nov., 1942. Wear index is number of thousands of revolutions required to wear the pile down to one-fourth of its matted thickness. The wear criterion of a carpet is D^2H, where D and H are density and height of pile, respectively. Wear per unit criterion is the quotient of the values in the other two columns.

Based upon experimental measurement of modulus, crimp leg length, back weight pressure, and fiber moment of inertia, the agreement between theory and results is grossly good, as shown in Tables I and II.

Reference to the work of Rainard and Abbott (page 301) reveals that the bulking properties of both single fibers and rovings are amenable to measurement and that the relationship between geometrical crimp and coverage can be partially determined by such measurement. Other geometrical considerations, such as the angle of the crimp and the leg length, are important but have been imperfectly defined to date.

Repeated laboratory measurements have shown in addition that there is an actual increase in wear for carpets made with crimped fibers over those made with straight fibers. This increase in wear occurs in spite of definitely lower single-fiber strength and elongation when the wear factors are measured by conventional testing methods (see Tables III and IV). The implication of this is that the real difference between the wools, such as the Indias, which are classified as tough, long-wearing, and good-covering, as opposed to the South Americans, which are easily crushed and show poor abrasion-resistance, is simply one of degree of crimp.

It has been experimentally demonstrated that the diameter of the roving bundle will go through a maximum at 7 to 8 crimps per inch (see Figure 5) and we can obtain this without production equipment. It has also been shown that certain configurations are most desirable from a wear standpoint and

TABLE IV. Comparison of Strength Characteristics of Crimped and Uncrimped Fibers Under Tensile Load

Characteristic	Crimped fiber Average	Crimped fiber Variation* (%)	Uncrimped fiber Average	Uncrimped fiber Variation (%)
Elastic limit	8,400 lbs./in.²	19	9,200 lbs./in.²	17
Breaking strength	16,500 lbs./in.²	20	39,400 lbs./in.²	20
Initial slope—load-elongation curve	544,000	33	1,019,000	16
Elongation at break	3.7%	56	30.2%	73
The fibers measured had the following geometrical characteristics:				
Length	2.23 in.	2.5	1.95 in.	4.9
Cross-sectional area				
Before breaking	2.08×10^{-6} in.²	43	2.00×10^{-6} in.²	25
After breaking	1.84×10^{-6} in.²	26	1.59×10^{-6} in.²	20

* Variation $= 100 \frac{\sigma}{\bar{X}}$ = Coefficient of variation.

these can also be obtained with the production equipment.

At first these seem like relatively impossible results, but closer examination of the mechanism of abrasion, as indicated by Backer [3], suggests that one of the problems in abrasion-resistance is essentially energy absorption over an expanded path. That is to say, for equal amounts of abrasive energy input the shorter the path over which the full load is applied the greater will be the abrasive wear. If a straight fiber is visualized as lying on a flat surface, the application of a shock load will cause energy absorption by direct compression and eventual collapse of the fiber. However, if that fiber were highly crimped and load was applied, the initial energy absorption must occur by mutual bending reactions since the work required to bend a fiber is relatively small compared with the work required to stretch a fiber. It seems reasonable to assume that the energy absorbed in bending reactions plays a small part. However, if the total number of bending reactions were greatly multiplied, as in the case of a highly crimped fiber, then the mechanism of absorption of energy of the impact load must be greatly different from that for a straight fiber. Such lateral loading, of course, is common in flat fabrics. In pile fabrics, a compressive loading on the pile ends is the significant factor where pile height is not too great. Again, however, energy is absorbed by bending, at least in the initial stages of impact. An uncrimped fiber can collapse under compressional load in only one or two places, but a highly crimped fiber should behave somewhat like a spring under compression. At the moment of this writing experimental work is in progress in our laboratory which aims to determine whether or not tests on flat fabrics will confirm the underlying supposition that energy absorption is of fundamental importance in abrasion-resistance.

TABLE V. Tensile Strength for Single Fibers (p.s.i.)

Boiling time (min.)	Relative setting time 0	1	7.5
0	28,900	17,150	9,640
1	18,350	25,600	14,300
3	31,800	21,000	17,000
5	18,300	22,000	13,650
20	22,200	26,200	16,300
60	29,800	16,650	12,900

Photomicrographs show evidence of the drastic changes which take place at the corner of the crimp as it is formed (see Figure 6). However, we do not believe that the major effect of crimp on the wool is fiber degradation, but that it is, rather, a change in geometry. Fiber strength is lowered because the shape produces a stress concentration in the fiber of a different order of magnitude from that of naturally crimped materials. Elementary stress analysis of the section shows that this may well be correct.

Assume a crimped fiber of uniform area A and radius r (see Figure 7), to which a tensile force F_0 is applied. The following is based upon simple beam theory.

The uniform stress on a section where the line of force coincides with the axis of the fiber is given by $S_0 = \frac{F_0}{A} = \frac{F_0}{\pi r^2}$. At a crimp the extreme fiber stress is given by $S = \frac{F_0}{A} \pm \frac{Mc}{I}$, where the plus sign is for the inside of the knee at the crimp and the minus sign

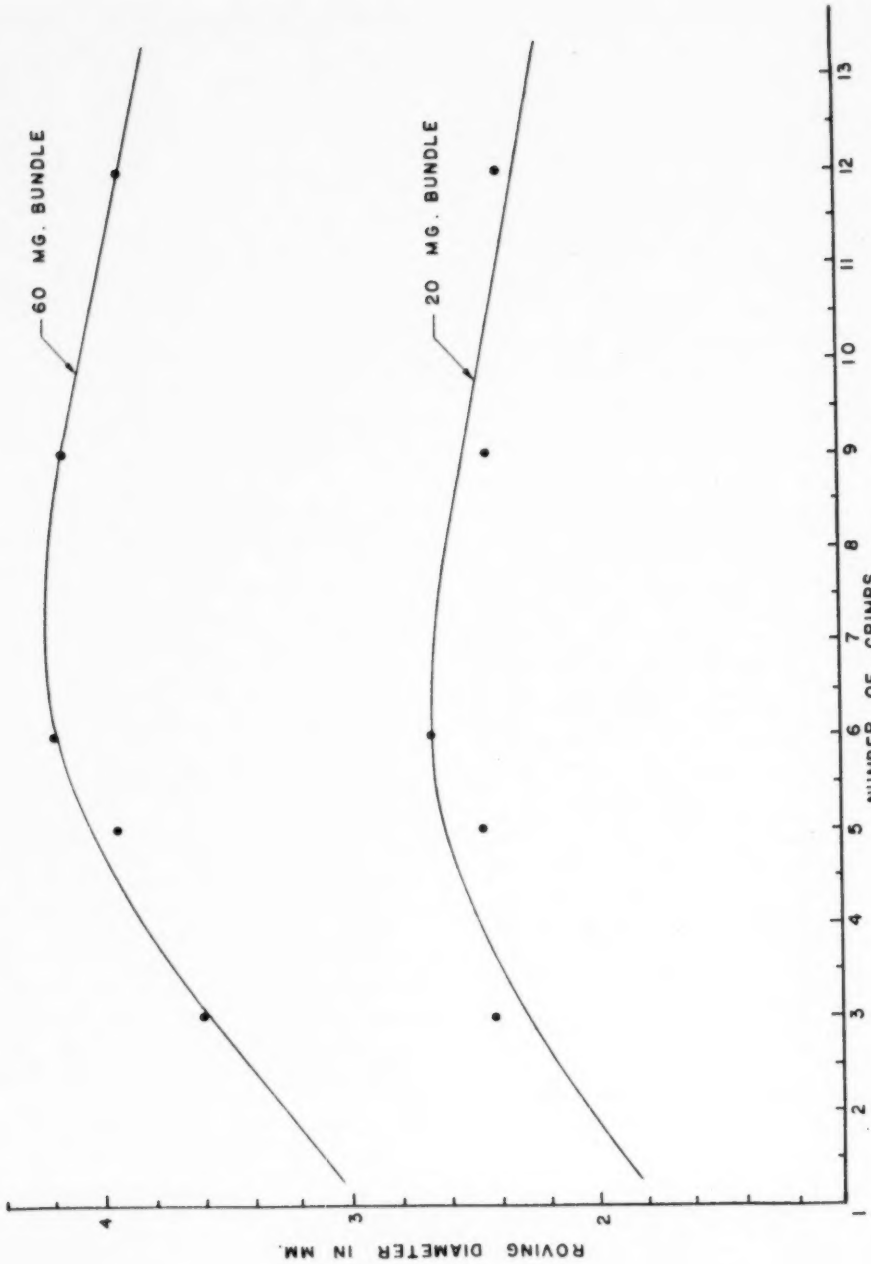

FIG. 5. *Wool roving diameter as a function of crimp frequency.*

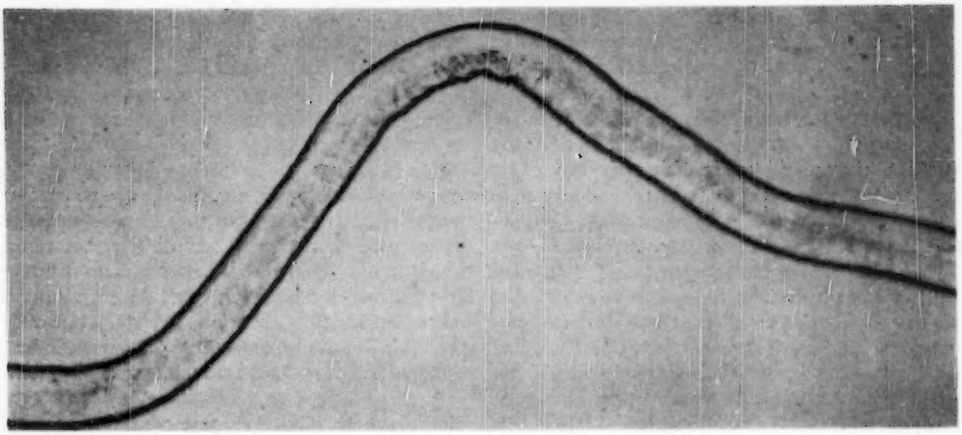

FIG. 6. *Photomicrograph of crimped wool fiber.*

is for the outside (only the plus sign need be considered for the maximum stress during tension).

Thus,

$$S = \frac{F_0}{A} + \frac{Mc}{I}; \quad S_0 = \frac{F_0}{\pi r^2}$$

$$M = F_0 X; \quad A = \pi r^2$$

$$c = r$$

$$I = \frac{\pi r^4}{4} \text{ for circular section.}$$

Let

$$X = nr;$$

then

$$S = \frac{F_0}{\pi r^2} + \frac{4F_0 nr^2}{\pi r^4}.$$

Therefore,

$$S = S_0(1 + 4n)$$

or

$$S/S_0 = 1 + 4n.$$

If S is sufficiently large to cause plastic flow, then the formula is no longer applicable.

For a series of selected examples the ratio S/S_0, or the stress concentration factor, was calculated to be in the range 10 to 400. Tests on crimped and uncrimped fibers showed the results listed in Table IV for strength reduction under tensile loads. As can be seen, there is a large decrease in the elongation of the crimped fiber at break—that is, the elongation at break was reduced from 30% for uncrimped fibers to 4% for crimped fibers, although the actual load at break was only cut in half.

It is possible to demonstrate that the effect of the crimping operation and the reduction in fiber strength are primarily due to geometry and not to fiber degradation. This is done by taking material which has been crimped—that is, the geometry changed—measuring the fiber strength, and then boiling this same material in order to release the fibers and to remove the crimp. When this is done fiber strength almost always increases, indicating that the reduc-

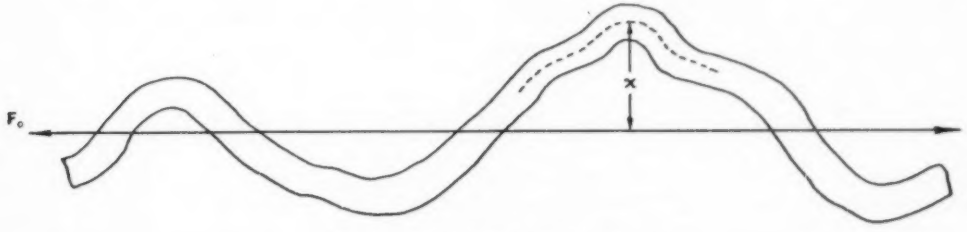

FIG. 7. *Crimped fiber under tension.*

tion in the amount of crimp present changes the geometrical situation; reduces the stress concentration at the crimp; and results in an increase in strength. Table V shows this quite clearly.

The other effect of the introduction of crimped wools instead of straight fibers into yarn is the change in slippage of one fiber past the other. This change might be expected to cause a decrease in yarn elongation at break over and above that obtained with uncrimped materials. Since this is not shown to be true, it must be concluded that when highly crimped materials are used there is sufficient fiber breakage before the slippage takes place to compensate for the interlocking effect of the individual fibers. Apparently, this kind of fiber interaction plays a tremendous part in determining the wear life of carpets [2].

It can be seen from the above data that there are two obvious characteristics which crimp imparts to a fiber: increased fullness and increase in wear life. When considering the problem of utilizing natural fibers, to date it has been considered that crimp is an inherent fiber property which could not be changed.

Conclusions

In effect, what this work attempts to point out is that the imposition of crimp on a normally straight fiber changes the mechanical behavior of the fiber, in many cases to a greater extent than could normally be predicted by ordinary study of single-fiber stress-strain properties. A fiber with no crimp reacts to load primarily in terms of tension or compression, with little of the mechanical behavior of the fiber influenced by the bending properties of the fiber. The imposition of crimp greatly exaggerates the bending properties of the fiber, and actually puts such fibers in a dominant position in certain types of fabric structures. For example, it can be seen that when top or roving is drafted the forces on the individual fibers rarely exceed those necessary to straighten out a crimped fiber. Yet in the drafting operation the bending modulus and the total number of crimps must clearly influence the force and energy relationships between fibers in any group fiber motion, such as that involved here. Friction remains as another significant factor, but related in its operating function to crimp. The entanglement of fibers either in felting or in spinning and a tendency for them to form stable structures rapidly obviously seem to be connected with crimp, and our condensation studies indicate quite clearly that a crimped fiber has a measurably higher ability to form stabilized structures. In terms of the fabric properties, the effect of crimp can seriously reduce the tensile strength which a fiber or yarn is capable of supporting. However, in terms of abrasive action due to lateral loading, or in terms of resilience of flat or pile fabrics, the impact of additional crimp is to influence greatly the energy-absorption characteristics of the fabric by multiplying by many times the number of bending reactions which the fibers must exert to the applied load. This not only can influence hand but also, as indicated previously, factors such as wear, coverage, and stability of the fabric. Although the work reported here was done on pile fabrics, preliminary studies on other types of fabric constructions indicate that the principles outlined may have equal importance in other fabric structures.

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Review of Research and Development in the Field of Textiles During 1949*

by

The Staffs of the Textile Research Institute and The Textile Foundation

I. Summary

This paper is a review of the literature published in 1949 relating to science and technology in the field of textiles. Because of the diversity of the subject matter, this summary, for reasons of brevity, must concern itself with only a few of the important contributions to the field.

One of the most interesting trends to observe in reviewing the literature in the textile field is the continued increase in applying to textile research those techniques and methods which have been developed in the general area of physical and chemical research. The study of fibers, yarns, and fabrics has been assisted materially by the use of new research tools, the development of which was accelerated during the last war. The new techniques made available to the research man in textiles have made it possible for him to undertake more refined studies on the structure of both natural and synthetic fibers. An increased activity in the study of natural fibers has been stimulated by the great strides made in the study and development of synthetic fibers. In fact, many workers have found it useful, and at times necessary, in the course of their studies on natural fibers to undertake research on synthetic materials in order to obtain a simplified and easily controlled environment for their work. Studies on polythene, polyvinyl chloride, and nylon have pointed the way to new techniques and ideas regarding structural configurations which have been fruitful in the research on natural fibers.

The fundamental study of the mechanism of fiber swelling and the connection between swelling and the dyeing characteristics of fibers have received attention. The use of radioactive tracers and of radioactive elements as activators in the dyeing process has been reported. It is anticipated that the tracer technique used with various dyes and dye bath constituents will permit a better understanding of the dyeing process and should result in the enhancement and closer control of quality, leading to more economical processing.

Studies on cross-bonding in natural and in synthetic fibers are continuing actively not only in the basic field of fiber-structure analysis but also in connection with the treatments of fibers to obtain dimensional stability and to protect them against degradation and chemical attack. The importance has been recognized of cross-bonds between molecular chains in cellulose and its derivatives, in keratin fibers, and also in synthetic materials. The study of such problems as swelling, dye penetration, crease-resistance and shrinkage, and the role of naturally occurring and artificially produced cross-bonds will not only add to our knowledge of structure but will also point to methods by which fibers may be modified to provide special properties.

The dynamic properties of textile fibers have received considerable attention during the past year. The behavior of the dynamic modulus and the manner in which a fiber dissipates energy at various frequencies have been examined both theoretically and experimentally. The energy loss by a fiber per cycle of its oscillation appears to have the same frequency characteristics as those for metals and rubber-like structures. This common behavior of textile materials, rubbers, and metals emphasizes the fact that the dynamic-loss characteristics must be identified with a mechanism which is not directly related to a given molecular structure. The relationship between the stress relaxation under static loading and a dynamic modulus and loss factor has been established; this relationship permits an estimation of the

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dynamic performance of a textile material from its static behavior. Ultrasonic techniques are taking a prominent place in the study of the physical properties of high polymers. The ultrasonic method holds promise of becoming more generally accepted as a laboratory tool for research as well as a practical method for obtaining the dynamic bulk properties of a material.

The flow characteristics of fluids form the basis of the viscometric method for determining the degree of polymerization of high-polymer molecules. Recently a method has been developed for evaluating the viscosity of a non-Newtonian fluid; this method has been applied to the study of the change in D.P. of cotton as it is modified by industrial processing.

In the field of instrumentation, effort has been directed toward the development of equipment for the study of the physical properties of fibers and yarns, for the use of radioactive isotopes in research and in plant operations, for control of quality and uniformity, for moisture and pH measurements, and for optical and spectrographic examination of materials. The chromatographic method of analysis used for the separation of constituents is finding wide application in the field of textile chemistry.

In the field of development, many of the published papers are concerned with the description of new equipment and of new processing techniques which have recently received plant trials. Here the problems relate to the automatic control of processes, to quality evaluation, and to plant operation. In connection with fibers and yarns, attention has been given to the processing and control of blends of fibers and to the processing of synthetic fibers on equipment originally designed for either cotton or wool. The effects of various chemical treatments on fibers, yarns and fabrics during processing are under active study, as evidenced by the number of papers describing the changes in textile materials resulting from such processes as scouring, bleaching, dyeing, and special treatments associated with stabilization. The dyeing of certain synthetic fibers continues to be a problem not only for a given material but also for blends with other synthetics or with natural fibers. Both crease-resistance and shrink-resistance are continuing to receive attention both from the point of view of the basic physicochemical mechanisms involved and from the point of view of industrial operations for producing crease- and shrink-resistant materials.

II. Introduction

The choice of material for this review has been dictated principally by the papers available in the scientific and technical journals published both in the U. S. and abroad and also in the standard abstract journals. The divisions of the review are somewhat arbitrary but reflect in large measure the distribution of subject matter throughout the fields of textile science and technology. Although it is believed that this review embraces most of the articles of consequence, there is no claim that it is absolutely complete. In fact, a sizable fraction of the material originally chosen for this review was jettisoned because the papers were considered to be inappropriate. Certain liberties in arrangement of the text have been made in order to emphasize either the research or the technological work. One of these liberties is the somewhat arbitrary division made between research and technology and also the sub-classification of the subject matter within a division.

The bibliography has been arranged to conform with the subject headings in the text. Within each principal division the references are arranged in alphabetical order by name of author; the items, however, are numbered consecutively throughout the entire bibliography.

III. Review Papers of General Interest

Brief reviews of textile research and development were given by Wright and Harris [28] for 1949, and by Goldberg for both 1948 [9] and 1949 [10]. A summary of cotton research prepared by the National Cotton Council of America [1] is a useful index of the current work in that field. Other reviews presenting the programs and results of research studies at The Shirley Institute [3], Textile Research Institute and The Textile Foundation [7, 15], the Southern Regional Research Laboratory of the Department of Agriculture [17, 23], and at the Wool Industries Research Association [25] are informative. Problems relating to the role of education in the textile field were discussed by Schwarz [16] and by Turner [21].

In the field of chemicals which bear on dyeing and finishing, discussions were presented by Boig and Fitzpatrick [4] on the chemical periodicals in the dye and textile industries, by McFarlane and Wilcock [13] on the dyeing and finishing of Fibro fabrics, by Sisley [19] on the use of compounds without active ions in the textile industry, and by White

[24] on the recent textile contributions by the organic chemical industries.

Another group of review papers includes those relating to the structure and properties of the fibrous state. Papers of a general character on the structure of textile fibers and the factors contributing to their usefulness as textile materials were written by Howlett [12], Moncrieff [14], Simmens and Urquhart [18], Staudinger [20], Urquhart [22], and Woods [26, 27]. Dicker [6] discussed the factors important to the resilience of fibers. Haigh [11] reviewed the properties, origin, and output of such specialty fibers as cashmere, camel's hair, mohair, and alpaca. The connection between plastics and the textile industry was considered by Giles [8], who pointed out that plastics may be used as components of a textile plant or machinery, as actual textile materials, as impregnating materials for textiles, or as coatings or surfacing materials on textiles. Buck and McCord [5] made a thorough review of the field of luster as it relates to cotton. This paper includes an analysis of the market for luster, together with a review of the technical aspects encountered in controlling and measuring luster.

IV. Research

A. Adsorption and Swelling

In this field the research studies were concerned with the adsorption of liquids by textile materials and the relationship between adsorption and the swelling characteristics. Preston and Nimkar [56] reviewed the swelling data for textile fibers and investigated the use of the centrifuge for measurements of volume swelling. White and Stam [61] studied the swelling of human hair and proposed a theoretical isotherm accounting for their results on adsorption and swelling. LeCompte and Lipp [53] investigated methods of testing for moisture in fibers. They concluded that toluene distillation is the most accurate method for wool. The water absorbency of fabrics was studied by Jackson and Roper [49].

Mellon, Korn, and Hoover [55] reported their water-adsorption study of wool, silk fibroin, and albumin fibers which had been treated to alter their internal structure. They concluded that such treatments do not influence water adsorption. Camposortega, Moncada, and Rowen [32] found no influence of light on the water adsorption of wool. Hutton and Gartside presented adsorption and desorption data for water on silk [48] and nylon [47]. Abbott and Goodings [29] considered the water adsorption of nylon also, and the density and swelling properties as well. Hall [39] discussed water adsorption and swelling for 10 rayon and synthetic fibers. Swelling and shrinking of artificial cellulosic materials in the form of fibers, yarns, and cloth were discussed by Girard [36, 37]. Swelling and shrinking of cellulosic materials were also reviewed by Tarkow [60]. Guthrie [38] extended the data on the integral and differential heats of sorption of cellulosic fibers. Campbell and Johnson [31] studied the adsorption of acetone by cellulose nitrate.

A theoretical treatment of the interaction of polymers and vapors (similar to the mixing treatment used for rubber) was suggested by Rowen and Simha [58]. Hill considered the statistical mechanics and thermodynamics of adsorption extensively from a theoretical standpoint. This series includes a treatment of the thermodynamics and heat of adsorption [43], the transition from adsorption to solution [46], unimolecular adsorption on nonuniform surfaces [42, 44], extension of the Brunauer-Emmett-Teller theory of adsorption [35, 45], and extensions of liquid theories to physical adsorption [40, 41]. Ross [57] compared the B.E.T. theory with the theory of Huttig.

A review of diffusion through cellulosic materials was presented by Stamm [59]. Kittelberger [52] gave an experimental and theoretical discussion of the diffusion of electrolytes through organic membranes. Diffusion through materials providing uniform capillary networks of molecular dimensions was discussed by Barrer [30]. Crank and Henry [34] reported the effect of a variable diffusion coefficient on the rates of adsorption and desorption. A general theory for diffusion and membrane permeability based on absolute reaction-rate theory was developed by Zwolinski, Eyring, and Reese [62]. Collins and Kimball [33] discussed diffusion-controlled reaction rates.

MacArthur and coworkers [54] showed that replacement of calcium ion by sodium ion in alginate fibers causes a contraction. King and Medley discussed the electrolysis of a keratin-water system [50] and the influence of temperature and adsorbed salts on the D.C. conductivity [51].

B. Dyeing

1. Properties of Dyestuffs and Dye Baths.—The color of the aminocoumarins was related to their

constitution by Hodgson [95] on the basis of the theory of resonance. Dufraisse and Loury [84] related the photo-oxidation of the non-nitrogenous vat dye Ixone to its structure. The structure of the diazoaryl sulfonates, sulfinates, and sulfones was discussed by Hodgson and Bailey [96]. Bowen [77] suggested that electron-transfer processes may be important in many of the photoreactions of dyes.

Weiss [119] considered the role of free radicals and radical ions in photosensitized reactions in solution. Evans and Uri [88] discussed the photochemical formation of atoms and radicals in solution. Hodgson and Norris [97] considered the mechanisms of diazotization and nitrosation. The photo-oxidation of azo dyes was considered by Desai and Giles [82] for aqueous solutions and by Blaisdell [76] for organic solvents. The theory of redox systems was applied to hydrosulfites and dye vats by Schaeffer [114].

Surface-active agents were found to be solubilizing agents for a number of currently used dyestuffs [67], and dextrine was found to stabilize reduced vat-dye solutions at elevated temperatures [69].

Contractor and Peters [80] prepared *tert*-butylphthalic acid and anhydride as new intermediates. Cupriferous dyes were reviewed by Krähenbühl and Wizinger [99], and vat dyes by Bender [75]. The colorimetric analysis of dye mixtures was discussed by MacAdam [105].

2. Theory and Chemistry of Dyeing.—Several reviews of the theories of dyeing and dye affinity appeared in the literature during 1949 [70, 93, 107, 115]. The diffusion of dyes in cellulose was reviewed by Morton [106].

Elöd and Fröhlich [87] examined the dyeing of polyamides with acid dyes. Experiments on the dyeing of viscose with direct dyes were reported by Preston, Mhatre, and Narasimhan [112], who studied the effect of swelling; and Standing and Warwicker [116], who studied the effects of carboxyl groups. Geake [92] presented data on the absorption of two vat dyes by cotton.

Peters and Speakman [110] developed the concept of "internal pH" for wool in acid. This concept was discussed further by Kitchener and Alexander [98] and by Peters and Speakman [109]. Donovan and Larose [83] gave data for the adsorption of sulfuric acid in the presence of sodium sulfate and discussed the application of the various theories of acid adsorption to their results. Lemin and Rattee [102] examined the leveling properties of acid dyes, and Gaunt [91] considered the theory of dyeing wool with chrome dyes.

3. General Papers on the Dyeing Process.—Elliott [86] reviewed methods of coloring textiles, and Teplitz [117] the methods of dyeing new synthetic fibers. A review of toners and organic fluorescent dyes was given by Petersen, [111].

The new Vat-Craft photochemical vat-dyeing process [66, 71] has attracted wide attention. Other new dyeing techniques, especially high-temperature processes [68, 94], were investigated.

The use of salt brine as an exhausting agent in dyeing was reported [63]; common salt was compared with Glauber's salt to determine the difference in rate of exhaustion and ultimate strength of direct dyeings on cotton and rayon [64]. Clark and McCleary [79] stressed the importance of the initial exhaustion rate in vat dyeing. The use of laboratory tests for assessing dyeing and leveling properties quantitatively were also examined [65, 103, 104].

4. Properties of Dyed Materials.—Royer [113] reviewed the application of microscopical methods to dyeing problems. A lively interest was shown in photochemical fading and degradation. Cooper and Hawkins [81], Vickerstaff and Tough [118], Lead [101], and Nordhammar and Gralén [108] evaluated the methods and variables involved in making light-fastness tests. Atherton and Seltzer [73] and Burgess [78] reviewed the fading of dyes. Egerton [85], Bamford and Dewar [74], Landolt [100], Ashton, Clibbens, and Probert [72], and Fox [90] examined photosensitization and tendering of textile materials.

C. Chemistry of Cellulose and Cellulose Derivatives

An interesting method for introducing disulfide cross-links into cellulose derivatives was studied by Izard and Morgan [144]. Substituent chloroacetyl groups were converted to Bunte salts, and these were oxidized in order to obtain disulfide cross-links. Reduction to the thiol form with thioglycollic acid was found to be possible. Nonreducible (methylenedithio) diacetyl cross-links were formed by the action of formaldehyde on the Bunte salt.

Timell continued his series of papers on cellulose reactions. In the formation of carboxymethylcellulose the primary hydroxyls react first, and in sulfoethylation the ratio of substituted primary and secondary OH groups decreases linearly with increasing degree of substitution [170]. He also compared [171] the accessibility measurement techniques of

Mark and Nickerson and found them to agree. Periodate oxidation of methylcelluloses prepared from sodium cupricellulose showed that etherification occurred first in the amorphous portions. By measuring the IBr absorption of highly substituted allylcelluloses [172] and assuming that the initial rapid reaction takes place in the amorphous region, Timell found that the ratio of amorphous to crystalline regions was the same in the fibrous ester as in the original cellulose (measured by periodate oxidation). Branched chain ethers can be prepared [173] if the cellulose is dispersed in quaternary ammonium hydroxide, which is used as a reaction medium and catalyst. In the most recent of his papers [174], he proposed the use of the change of rate in the reaction of cellulose with sodium in liquid ammonia to measure the degree of accessibility of the cellulose.

Stakheeva-Kaverzneva, Ivanov, and Salova prepared 6-desoxycellulose via tosylcellulose, iodotosylcellulose, and unsaturated desoxycellulose [166]. Seven possible reactions between cellulose and formaldehyde were reviewed and discussed [120].

Two papers [126, 127] by Entwistle, Cole, and Wooding reported studies on the kinetics and mechanism of the autoxidation of alkali cellulose. The kinetics of the uncatalyzed reaction indicated that it proceeded by way of chain mechanism involving transient free radicals. Substances which generate free radicals were shown to catalyze the reaction, while silver and gold compounds, as well as typical organic antioxidants, inhibit it. Possible depolymerization processes were discussed in the light of these results. McBurney and coworkers [128, 129, 147] studied the light and heat stability of ethylcellulose and the heat stability of cellulose acetate. The heat degradation of both materials is an oxidation reaction in air. The same type of oxidation occurs when ethylcellulose is exposed to ultraviolet light, but the rate of reaction is much higher. The commercial manufacture of oxycellulose by the nitrogen-tetroxide method was described by Kenyon, Hasek, Davey, and Broadbooks [145]. Chemical and physical specifications for its use as surgical gauze were listed. Nevell [152] also reviewed the oxidation of cellulose with nitrogen tetroxide.

The photochemistry of cellulose was studied by Launer and Wilson [146]. In the far ultraviolet region, water retards the deterioration of cellulose, whereas oxygen has no effect. In the near ultraviolet region, the reverse is true. This difference is assumed to be due to the fact that photons in the far ultraviolet region have sufficient energy to break C—C or C—O bonds, whereas in the near ultraviolet region they can induce reaction only if oxygen is present. The effect of water vapor is interpreted as indicating that part of the water is chemically bound to the cellulose. In the far ultraviolet region about one in each thousand photons absorbed causes chain scission. A group of Indian workers [153] showed that the changes taking place in fabrics which have been stored in atmospheric oxygen after having been exposed to ultraviolet radiation can be brought about immediately by boiling in 1% NaOH.

The structure of cellulose continues to receive attention. Van der Wyk and Studer [175] concluded that pure cellulose has no free carboxylic acid groups, but that its weak acid nature is due to an accumulation of OH groups in the glucopyranose residues. Steele and Pacsu [169] described a technique for studying the infrared spectra of regenerated cellulose films. Dickey and Wolfrom [125] subjected cellulose to mild acetolysis and chromatographically separated from the product a complete polymer-homologous series of sugars from α-*D*-glucopyranose pentacetate to α-cellohexose eicosacetate. An interesting series of papers by Reeves [154–156] describes studies of cuprammonium glycoside complexes which may eventually throw some light on the mechanism of cellulose solution in cuprammonium.

Heterogeneous acid hydrolysis of regenerated cellulose was shown by Hermans and Weidinger [137] to increase the crystallinity 39% to 49%, as determined by Hermans' x-ray method. However, ramie did not undergo any significant change. Mehta and Pacsu [148] subjected native cellulose and rayon to heterogeneous methanolysis, using 43% HCl in methanol. As in mild hydrolysis, the limiting D.P. for rayon is ~ 60, for cotton ~ 250. The product of methanolysis was hydrolyzed, and the rate of increase in reducing power was found to be many times faster than that of normal methyl glucopyranosides. This was interpreted by the authors to mean that the product corresponded to the dimethyl acetal of an open-chain sugar. Mehta and Pacsu [149] also examined the degree of polymolecularity of acid-modified celluloses. They fractionated nitrates of hydrocellulose from native cotton, surgical cotton, and viscose rayon. Their results indicate that a large proportion of the hydrocelluloses are uniform in that almost 33% of the methanolyzed native cotton consists of molecules with D.P. 252, about 70% of the

hydrocellulose from surgical cotton has D.P. 260, and some 36% of the material from viscose rayon possesses D.P. 62. These results are interpreted on the basis of the new concept of cellulose structure proposed by Pacsu. Harris and Kline [136] investigated the destructive hydrolysis of wood cellulose with hydrochloric acid and with sulfur dioxide. The latter has a rate constant for the reaction equal to 1/7 that of hydrochloric or sulfuric acid under similar conditions and yields less reducing sugar.

Hessler and Merola [138] compared several methods for determining the cellulose content of bast fibers. The reagent they proposed consists of one part monoethanolamine and three parts ethylene glycol. Conrad and coworkers [164] made a detailed study of the determination of cellulose by acid-dichromate oxidation. Failure to account for the theoretical amount of cellulose in this procedure was found to be due to retained moisture in the sample and to the formation and loss of some carbon monoxide. An "experimental" equivalent weight, taking into account these discrepancies, was calculated for routine use. Hamlin [135] investigated the wet ashing of textile materials, using a mixture of nitric, sulfuric, and perchloric acids, and found in many cases that this procedure is superior to the dry methods normally employed.

The conversion of cellulose into viscose by xanthation was the subject of a number of papers. Mitchell [150] followed changes in D.P., α-cellulose content, carboxyl content, and pentosan content of the cellulose from ageing through spinning for several pulps of different initial purity. Special consideration was given to depolymerization reaction. Some effects of time, temperature, and oxygen concentration on the extent of alkaline degradation were shown. The presence of oxygen may be the factor determining which of two types of alkaline degradation will occur. Samuelson [160] described a technique for comparing the reactivity of wood pulp toward carbon disulfide. The samples were xanthated with carbon disulfide emulsified in NaOH solutions of varying strength, and the undissolved residue was determined. The amount of residue depended little on the molecular weight and viscosity of the cellulose, but mercerization, ageing in air, or hydrolysis by HCl increased it because of changes in the internal structure of the fiber. Scherer and Phillips [163] fractionated a cellulose xanthate derivative and found it to be heterogeneous both in chain length and in degree of substitution. The assumption that this heterogeneity is a result of the slow penetration of the reagent into the soda-cellulose structure appears to be in agreement with known types of topochemical reactions. Danilov and his coworkers published three papers on the chemistry of xanthates and viscose. The first [122] showed that the oxidation of the alkali cellulose is sharply retarded by Na_2SO_3 or its mixture with Na_2S. The latter alone is less effective. The action of these salts is complex in viscose solutions since they are also sulfur-removing agents. The second and third papers [123, 124] are concerned with components of viscose which add or lose an atom of sulfur, and with thioanhydrides of xanthic acids and their transformations.

Fordyce and Simonsen [130] studied the evaporation of binary mixtures of solvents from cellulose ester solutions. In the absence of cellulose derivatives, the solvent mixtures evaporate primarily according to liquid-vapor equilibrium characteristics; in the presence of cellulose esters, evaporation in many cases deviates from this behavior, showing selective retention of certain types of solvents. Fortess [131] used specific-gravity measurements to detect the interaction of various organic liquids with cellulose acetate fibers. The rate and extent of penetration was determined for a large number and variety of pure organic liquids. The maximum apparent specific gravities for the yarn were found to be functions of the polar groups on the molecules of the liquid medium, as well as of their size and shape. Rosset and Paris [159] deacetylated cellulose acetate homogeneously and observed the changes in its properties as the reaction proceeded. Affinity for direct dyes showed a maximum at 26% acetic acid content. Water absorption and swelling have maxima at 2% and 13%, respectively. Density increases regularly with increasing acetyl content. Howlett and Urquhart [143] found that traces of oxalic acid introduced into cellulose acetate fabrics during manufacture are the cause of the deterioration of the fabric when normally washed, and also have a solubilizing effect when the fabric is stored in a climate of high humidity. Hamalainen and Reid [134] partially acetylated cotton cellulose with ketene, using perchloric acid as a catalyst. Samples containing up to 17% acetyl retained their fibrous structure and suffered only slight degradation. The acetylation was accompanied by the formation of yellow and brown ketene polymerization products. Roberts [158] investigated the use of diphenylamine as a color reagent for detecting the presence of nitrates in cellulose esters.

For general use, 0.1 g. of diphenylamine in a mixture of 100 ml. of conc. H_2SO_4 and 30 ml. of H_2O is satisfactory. Quantitative estimates are possible when films of known nitrate composition are used for comparison.

Badger and Blaker [121] investigated the form of nitrocellulose molecules in solution by light-scattering methods. The molecules are relatively stiff and are shown to be nearly fully extended up to a D.P. of about 100. Above this D.P. the molecule begins to assume the properties of a random coil. Scherer and Masuelli [162] studied the relationship between solubility and D.P. for cellulose nitrate. The solubility is a linear function of the logarithm of the D.P., which is in agreement with the relationship found with other high polymers. Reid, Mazzeno, and Buras [157] investigated the composition of two types of cellulose phosphates. Phosphates prepared by the urea-phosphate method have all their phosphorus in the form of a monosubstituted phosphate ester. Those prepared by the action of $POCl_3$ and pyridine are similar, except that in a typical sample 23% of the phosphorus can be accounted for in the form of a disubstituted phosphate ester.

Steele and Pacsu [167, 168] made some studies on trimethylcellulose. Heterogeneous hydrolysis of this material in weak HCl showed a rapid drop in D.P. to a limiting value of 60. The apparent rate constant, assuming first-order reaction kinetics, changes rapidly at first but more and more slowly as the D.P. falls from 900 to 60. The behavior of partially methylated cellulose on mild heterogeneous hydrolysis confirmed the topochemical nature of the alkylation reaction. Attempts to fractionate trimethylcellulose from $CHCl_3$ or pyridine solutions resulted in difficulties which indicate that neither solvent is suitable for fractionation. The difficulties are related to the solubility behavior of trimethylcellulose, which is discussed in the light of present theories of polymer solubility. McBurney [147] studied the oxidative stability of ethylcellulose at elevated temperatures. The rate of oxygen adsorption and length of the induction period are not functions of the degree of substitution or viscosity. Initial attack of the oxygen is to form peroxides; once formed, the peroxides decompose at a rate faster than their formation; thus there is a maximum in the peroxide content. Scherer and McNeer [161] investigated the fractionation of ethylcellulose from various solvent-nonsolvent systems. Best results were obtained by adding an acetone-water mixture to a solution of the material in a mixture of acetone and ethyl acetate.

Higginbotham and Morrison [141] determined the conditions under which amylose and amylopectin may best be distinguished by iodine absorption, and used this technique in studying [142] whether or not corn starch and some root starches can be separated quantitatively into the above two components. Higginbotham later showed [139] that the absorption of iodine by amylose does not reach a limiting value other than that imposed by the limited solubility of the iodine, although he confirmed the fact that the absorption occurs in two stages. He showed further [140] that amylopectin absorbs as much iodine as amylose if the iodine concentration is sufficiently high. Myrbäck and Sillén reported experiments [151] which indicate that the irreversible process of forming a polysaccharide from glucose-1-phosphate by two enzymes (one forming 1,4-, the others 1,6-linkages) results in amylopectin, while the corresponding reversible synthesis produces glycogen. Semmens [165] observed that polarized infrared radiation hydrolyzes potato starch, and suggests that this may be the physical mechanism of enzymic hydrolysis. French, Levine, and Pazur [132] prepared and characterized amyloheptaose by controlled hydrolysis of Schardinger's β-dextrin. Haller [133] suggested that the name "soluble starches" be restricted to those having a specific group of chemical properties.

D. Viscosity, Molecular Weight, and Related Studies

Rivlin [191] and Oldroyd [190] developed mathematically the hydrodynamics of the flow of non-Newtonian fluids through tubes of circular cross-section. Brinkman [181] surveyed various problems related to the hydrodynamic properties of swarms of particles and of macromolecules whose flow can be treated by extending the hydrodynamic equation with a damping term which is proportional to the mean rate of flow. Krieble and Whitwell [188] derived an equation for the calculation of the counterpart of viscosity for a pseudoplastic or a dilatant liquid and have shown how it would be useful in measuring the D.P. of high polymers. Wilson [195] proposed a formula relating the intrinsic viscosity of a linear polymer to its molecular weight and structure. Intrinsic viscosity is given as a function of the number of links in a polymer chain, and two constants, K_1 and K_2, are expressed in the terminology of the Kirkwood-Riseman theory. Vand [193] extended the

theory of viscosity of suspensions to cover the molecular particle-size range by introducing a correction factor containing the ratio of the volumes of the solvent to the solute molecules.

Davis and Elliott [184] reviewed and summarized the points which are of importance in making rapid and accurate determination of intrinsic viscosity, at the same time obtaining as much additional information as possible. Howlett and Belward [187] suggested two methods for rapidly determining the cuprammonium fluidity of cotton at 0.5% concentration. Stow, Horowitz, and Elliott [192] adapted the rising-column capillary viscometer to the high-viscosity measurement range and analyzed its operation. Results agree with falling-ball techniques when properly designed instruments are used.

Fox and Flory [185] measured the intrinsic viscosity of some polyisobutylene fractions over a wide range of temperatures and in a number of solvents. The results agree with the postulate that intrinsic viscosity reflects directly the effective volume of the molecule in dilute solution, the hydrodynamic factor expressing the interaction of the equivalent sphere with the solvent remaining very nearly constant, except perhaps at very low molecular weights. Cragg and Simkins [183] also reported data which show clearly that solvent effects on the intrinsic viscosity are strongly dependent on molecular weight for linear flexible polymers, and they support the concept that the differences in the intrinsic viscosity of such a polymer in a good or poor solvent is due primarily to differences in the tightness of coiling of the molecules in solution.

Beall [177] made a study of the possibility of improving the arithmetic treatment involved in the determination of the distribution of the molecular size in a sample of cellulose. The process considered was fractionation by successive precipitation. Beauvalet and Clement [178] also presented a mathematical approach for determining D.P. and the distribution curve. Fractionation was used in verifying the theory. Aggarwala and McCarthy [176] measured the distribution of molecular weights in bamboo cellulose, and Wannow and Thormann [194] studied the chain-length distribution for oxidized cotton. Complex frequency distributions were observed in both cases.

A study by Cleveland and Kerr [182] on the osmotic pressure of amylose acetate fraction indicated that these derivatives are associated to different extents in organic solvents; the extent of association appears to depend on the solvent. Meyer and coworkers [189] examined starch solutions and pastes and interpreted their properties on the basis of the physical-chemical behavior of the individual components of the starch granule. Bechtel [179] and Bechtel and Fischer [180] described viscometers which are capable of obtaining satisfactory rheological data on starch pastes for either industrial testing or research purposes.

Herrent, Mouraux, and Lude [186] studied the viscous phenomena associated with concentrated colloidal solutions of cellulose xanthate which are important in rayon manufacture.

E. Deterioration of Materials

York [221] and Kornreich [205] independently reviewed the sources and causes of damage to textiles during manufacture and use, including methods for identifying and determining the extent of some types of damage. Murdison and Roberts [212] studied the effects of laundering and storage on cotton cloth. In the course of 100 launderings in an automatic machine progressive wear effects on the fabric were demonstrated. Tensile strength fell steadily; fluidity increased only after 50 washings. The Congo red test indicated gradual increase in mechanical rupture of the cuticle. Samples retested 8 years after laundering showed higher fluidity and lower tensile strength at all washing levels. Race [216] discussed the degradation of cotton during atmospheric exposure, with particular attention devoted to the theories which would explain the post-irradiation reaction of cellulose with oxygen. Bogaty [199] found that cotton fabrics which had been treated with copper naphthenate deteriorate more rapidly on outdoor exposure than untreated fabrics. Free copper sulfate was found to accelerate the tendering. Perti, Ranganathan, Subramanian, and Sud [214] examined the role of metals more extensively and agree that copper, as well as tin and titanium, accelerates degradation. Manganese retards deterioration markedly, while iron and aluminum show some protective action. Chromium has no effect. Derrett-Smith [202] described a modification of the solubility-number test for measuring cellulose degradation.

Appleby [196], in an extensive review of the literature on the effect of light on textile materials, considered the influences of the light source, atmosphere, and physical state of the sample. Lowry [208] proposed that the stability of various colored ma-

terials to light be specified by spectrophotometric examinations before and after exposure. He discussed various methods for interpreting the data. Egerton [203] studied the action of light on acetate rayon and nylon dyed with various dyes. The photochemical degradation of both of these materials, dyed or undyed, was found to be due to oxidation processes. Acetate dyes which are effective in increasing the degradation of nylon were found to be capable of increasing the degradation of undyed cotton irradiated in their vicinity. Pinte and Millet [215] reviewed the influence of the principal destructive factors on the photochemical degradation of dyes.

Callow and Speakman [200] studied some of the chemical and physical changes which take place when jute is exposed to air and light. All of the main components of jute were found to undergo degradation. Methylation and acetylation under proper conditions will reduce the discoloration that jute suffers on exposure. Chamberlain, Lucas, and Speakman [201] found that calcium alginate fibers undergo rapid and severe degradation due to chain scission during exposure to light. The mechanism of the breakdown could not be deduced from their experiments. Chromium alginate was found to be much more light-resistant.

Marsh and coworkers [210, 211] surveyed the literature on the occurrence of fungi which cause cellulose deterioration and in addition tested some 400 fungus isolates for cellulose-decomposing activity. Activity is very widely distributed but is not universal. Siu, Darby, Burkholder, and Barghoorn [218] studied the mechanism of the growth of 11 species of microorganisms on cellulose and other substrates. One firmly bound substituent in each anhydroglucose unit was sufficient to prevent microbiological attack, whatever the nature of the group. Block [198] reported some outdoor exposure tests on cotton duck which had been treated with various fungicides. Copper, silver, and phenolic compounds stood up well after 2 years in the field. The copper ion rather than the anion or molecule appeared to be the effective agent in copper compounds (*cf.* above). Mercurials and other heavy-metal fungicides did not afford 2-year protection. Marsh [209] tested some fabrics with organisms having a high tolerance for copper and mercury, which renders fungicides which are dependent on these metals less effective. Several specific treatments for mildewproofing fabrics have been described: "Sanitizing" by Williams [220], phenyl mercuric "Fixtan" by Hopf and Race [204], and nitrosophenylhydroxylamine by Bayley and Weatherburn [197]. Partridge and Key [213] also discussed some of the commercial processes for rotproofing fabrics.

LaFleur [206] pointed out that wool damage may be roughly classified into hydrolysis, reduction, oxidation, and incipient decomposition caused by exposure to light or heat in the presence of air. Data were presented showing the changes in wool during laboratory treatments which simulated these various kinds of damage. Stahl, McQue, Mandels, and Siu [219] studied the microbiological degradation of wool, particularly sulfur metabolism. Inorganic sulfate was found to be a product of the decomposition of wool by fungi. Shema and Appling [217] reported that the degree of bacterial degradation of woolen felts can be determined by the extent to which the fibers absorb safranine. Lindberg [207] observed in electron micrographs thin membranes about 100 Å. thick on the outer surface of wool. This is the osmotic membrane which is observed in Allwörden's reaction. A reaction depending on such a fragile layer is too sensitive to be used for estimating damage. Whewell and Wood's test for mechanical damage and the Kiton Red G test for extent of chlorination have a similar high sensitivity because of this membrane.

F. Structure and Properties of Cellulose Fibers

The excellent series of reviews on fiber structure in 1949 by members of the Textile Institute included, among others, papers on cotton [228], flax [259], long vegetable fibers [260], and viscose rayons [251]. In addition, Berkley [224] reviewed variations in the structures of natural cellulose fibers which make them useful in various applications. A brief description of the Brazilian caroa fiber was also published [230].

Interest in jute as a commercial fiber is increasing, as shown by several papers on moisture relationships and tensile strength of jute [227, 247, 252, 253].

The bulk of research on native cellulose fibers reported in 1949 was concerned with their fine structure. Techniques used by various investigators included x-ray methods for crystal structure [240, 245], crystallinity [233, 234] and lateral-order determination [232, 237], electron micrography [246, 254], density measurements [262], moisture sorption [238], and reaction rates [235, 244]. Electron

microscopy has revealed a network arrangement of fibrils in the primary walls and a more or less parallel arrangement in the secondary walls of native fibers [246]. The primary wall encases the fiber, retarding the entrance of reactants with the cellulose within the fiber. Milling the fibers to disrupt the primary wall results in increased penetration and higher reaction rates [235]. This observation is confirmed by density measurements on whole and cut cotton fibers immersed in wetting, nonswelling liquid media [262]. Density studies of cottons in various immersion media indicated the presence of small pores or void spaces within the fiber [262]. This has been substantiated by the nitrogen-adsorption studies of Hunt, Blaine, and Rowen [239] on cotton linters, in which evidence of pores 16 to 20 Å. in radius was obtained.

Considerable research on the structure and properties of viscose rayon was reported during 1949. Bredée [226] reviewed the characteristics of various rayons and their methods of manufacture. Structure of skin and core received special investigations [250], one of these utilizing the unique method of successive topochemical acetylations and removal of outer layers with solvents in order to study the swelling characteristics of the various parts of the fiber [229]. Hermanne [231] published a thermodynamic interpretation of the mechanical behavior of rayon.

The relationship between mechanical behavior and molecular structure appears to have received much less attention than the subject deserves [241, 242, 256]. Some work on the analysis for minor constituents in native cellulose fibers is still being reported [243, 248].

The Goldthwait differential dye test for cotton fiber maturity is being used more and more for routine cotton quality evaluation [222, 261]. Armfield and Boulton [223, 225] investigated the theory and application of this test to both cotton and viscose rayon and concluded that the test can be used to differentiate between coarse and fine rayon fibers. Work [264] showed that unstretched, swollen cellulose acetate dyes green in the test, whereas the stretched and oriented fibers dye red. Sondhelm [257] showed that thin cotton fiber tips generally dye green even when the center portion and base of the fibers dye red. Heyn [236] proposed a method for determining the maturity of cotton by chemical analysis based on wax content of the fibers.

The production and properties of cellulose acetate were discussed in the excellent reviews by Wilson [263] and by Work [264]. Secondary cellulose acetate fibers as spun have a definite crystallinity which cannot be increased because of steric effects. Swelling and stretching, however, produce orientation which can be maintained during hydrolysis to produce highly crystalline and well-oriented cellulose fibers [249]. The cellulose acetate fibers themselves have a strained outer layer which shows cracks and other defects after being stretched [255].

G. Structure and Properties of Protein Fibers

1. Structure of Wool.—The fine structure of wool was reviewed [271, 272]. Barker [279] reviewed work on the fibril structure of wool, and Hopkins [301] considered the chemical, morphological, and physical properties of wool fibers. Consden [285] and Zahn [329, 331] gave reviews of the chemical structure of wool. The use of x-ray diffraction techniques in studying crystalline proteins was discussed by Perutz [320], who also outlined the use of Patterson's method for interpreting x-ray diffraction patterns of crystalline materials. Lindberg and coworkers [306] gave an account of the fine histology of wool fibers and hairs as revealed by recent optical and electron-microscope studies. In this work they found evidence for the multiple structure of the cuticle. The authors have shown the practical importance of the fine structure of wool in dyeing, damage tests, shrinkproofing treatments, and surface friction.

Ambrose and coworkers [269, 270], using polarized infrared techniques, found evidence for chain folding in both synthetic polypeptides and in keratin; their results may be explained if the peptide chain is folded and maintained in the folded state by intrachain hydrogen bonding. A spiral structure in a closely packed system does not appear to them to be consistent with the α-β transformation.

Darmon and Sutherland [286], in discussing this work, stated that because of the existence of two or three types of hydrogen bonding in proteins, the model for the structure of α-keratin proposed by Ambrose and Hanby is probably an oversimplification of the actual structure. Astbury [276] pointed out that the type of folding suggested by Ambrose and coworkers would not give the proper x-ray diffraction pattern for the α-keratin, that this structure is not sufficient to account for an extension of from 35% to 40%, and that at first sight the structure of certain synthetic polypeptides bears a strong resemblance to the α-pattern of native keratin even

though there is an enormous difference between the properties of the synthetic polypeptides and those of the natural fibrous proteins. Astbury [275] changed the proposed structure of polyglycine on the basis of a suggestion made by Bunn that polypeptides with shorter side-chains might be expected to fit together in approximate close arrangements like those found in polyamides (nylon). The revised structure brings the calculated and the measured density into agreement.

The rubber-like elastic properties of fibrous proteins were discussed by Astbury [274] and Meyer [315, 316] in terms of the current structural models for these fibers. Jagger and Speakman [303] gave a brief account of investigations on the relaxation and recovery of animal fibers and the mechanism of restrengthening. They found that arrangement of the structure which had been disorganized by stretching is accompanied by a restrengthening which proceeds slowly in water at low temperatures but which can be accelerated by raising the temperature. These results indicate that the beneficial effects of ageing processes, which are now conducted at room temperature, may be obtained in much shorter periods of time by raising the temperature.

In studying the stress-strain behavior of Lincoln wool, Ripa and Speakman [321] showed that several successive 30% extensions in water and in 0.1*N* hydrochloric acid can be given to these fibers without serious change in their elastic properties. This confirms results obtained on other wools and on human hair.

In studying the surface properties of wool, Lindberg [207] reported the presence of a thin membrane (epicuticle) on the surface of the wool fiber; the effect of such a membrane on damage has been mentioned earlier in this review in connection with the deterioration of textile materials. In Lindberg's experiments wool was given a slight chlorination, then shaken with water, and a drop of the suspension was examined in the electron microscope. A large number of membranes about 100 Å. thick were observed. These are considered to be the osmotic membranes which are responsible for the bubbles and sacs formed in Allwörden's reaction. It was pointed out that the surface membrane plays an important part in the diffusion of dyes and other large molecules into the wool fiber. Mercer, Lindberg, and Philip [314], continuing their studies on the structure of the cuticle, showed that three components may be recognized in the cuticle of wool and hair: endocuticle, exocuticle, and epicuticle. By dissolving the keratin, it is possible to separate the epicuticle, thickened by the adhesion of more or less of the intermediate layer, in the form of a continuous tube, the external surface of which is that of the original fiber.

Lindberg and Gralén [304, 305], in further studies on the surface properties of wool fibers, examined fiber-to-fiber friction, using a fiber twist method developed by them. The study of single-fiber frictional properties as influenced by oxidizing and reducing agents indicated that chlorination first increased both anti-scale and with-scale friction, but with increasing severity of treatment the scales were attacked and were then easily removed by abrasion. Potassium permanganate produced an increase in both frictional coefficients but did not destroy the scale substance, except when followed by an after-treatment with sodium bisulfite. The authors concluded that the frictional behavior of wool and its change when treated with different reagents can be explained by the histological structure of the outer layers of the fiber. Mercer [310, 313] applied x-ray and birefringence techniques to study the orientation and hardening of keratin in the hair follicle and also used the electron microscope to study wool in which polymethacrylic acid had been polymerized. The polymer seemed to be deposited chiefly in the extra-fibrillar regions of the wool fibers.

Evans and Gergely [290] discussed the possibility that bands of energy levels exist in proteins; these banded electronic energy levels may confer a small extra stability to the protein structure. Examination of the photoconductivity and diamagnetic anisotropy should reveal the correctness of these assumptions.

2. The Chemistry of Wool.—A large segment of the published research on the chemistry of wool is concerned with reactions involving the disulfide linkage. The reaction of wool with dilute solutions of permanganate at a pH of less than 2 was studied by Alexander, Carter, and Hudson [267] as a non-felting treatment; the principal reaction is an oxidation of the disulfide bond, with the reaction controlled by diffusion. The reaction kinetics of wool with various solutions was reported by Alexander, Gough, and Hudson [268], who showed that the rate-controlling factor in the reaction of a solution is either diffusion through a liquid film or diffusion in the fiber, the activation energies being 6 and 12 kg.-cal./mole, respectively.

Elöd and Zahn [289] discussed the chemical damage to wool by dithionite during printing. This

causes the cystine bonds to break, resulting in supercontraction at 100°C. In order to make the wool more resistant to the damage caused by discharge printing, the wool or hair is first reduced by dithionite at 40°C; this treatment is then followed by alkylation with alkali dihalides, such as dibromoethane, in order to produce new bonds from the SH groups present through reduction. Elod and Zahn [288] believe that supercontraction of animal hairs and wool may be due to a kind of "melting" process which occurs within the keratin fibers rather than to the breaking of cystine cross-bonds. By treating animal hairs with phenolic solutions, such as phenol, resorcinol, catechol, and pyrogallol, the peptide chains were free and able to contract. A series of contraction studies on polyamides and natural silk in these phenolic agents were made. The results are of interest since these fibers do not have cystine cross-bonds, although their behavior is similar to that of animal hairs at pH 1.5 to 5.0 in phenolic solutions. These supercontractive effects may be applied to study the changes in linkages during wool finishing.

Reactions involving the disulfide linkage, principally those used for resistance of wool to shrinkage or to permanent set, were reported by Speakman and coworkers; reactions of the cross-bonds with alkalies or cyanides [293], chlorine, sulfuryl chloride and sodium hydroxide [291, 292], 3:4-isopropylidene-1:2-5:6-dianhydromannitol [283], and allyl isothiocyanate [319] were reported.

Alexander [265] pointed out the role of hydrogen bonds in supercontraction and permanent set. In addition to the breaking of disulfide linkages, the breaking of hydrogen bonds with strong aqueous solutions of bromide prevents supercontraction in a boiling solution of bisulfite; however, the fibers still contract in LiBr solution. Alexander stated that both supercontraction and relaxation of permanent set can be effected without breaking covalent sulfur bonds.

Elod and Frohlich [287] studied the effect of both strong and weak acids on wool, silk, and polyamides. The absorption of strong acids by wool and natural silk is supposed to reach a maximum at pH 1.3, remaining constant with increasing acid concentration. However, above pH 0.8 the absorption of acid increases but no damage is done to the protein fibers. The increase of acid absorption may be due to the take-up of protons by the negatively charged oxygen of the peptide group. Weak acid take-up differs from that of strong acids because the absorption increases with a decrease in pH. Wool, natural silk, and polyamides behave similarly when treated with both strong and weak acids.

Fraenkel-Conrat and Mecham [296], in a study of the reaction of formaldehyde with proteins, reported that formaldehyde treatment at pH 3 to 8 at room temperature results in an increase in molecular weight, as determined by osmotic pressure. The increase in molecular weight is ascribed to the formation of methylene bridges between the amino and other reactive bonds of two or more molecules. It was found that proteins or polypeptides which are rich in amide but poor in amino groups are not readily cross-linked as a result of this formaldehyde treatment. Middlebrook [318] examined the reaction of formaldehyde with proteins under acid conditions at 100°C and found evidence of combination; some of the resultant reaction products can be removed by distillation in acid. For wool and human hair it is postulated that the aspartic acid is in the form of asparagine residues, and the glutamic acid is in the form of glutamine and glutamic acid residues. The formaldehyde is thought to form cross-bonds between the glutamic amide groups and some other groups, including the guanidino groups of arginine; the formaldehyde cross-bonds can be broken by distillation in dilute phosphoric acid. Formaldehyde probably combines with the asparagine amide groups to form 6-hydroxytetrahydropyrimidine-4-carboxylic acid; this is stable to boiling dilute phosphoric acid.

The formation of polymers in wool, for purposes of stabilization, was discussed by Lipson and Speakman [308] for the case of methacrylic acid and methyl methacrylate polymerized internally, and by Lipson [307] for vinyl polymers; Alexander, Carter, and Earland [266] discussed the surface treatment of wool fibers with silicone plastics to obtain nonfeltability.

The factors affecting the formation of keratin during the growth of hair was studied by Mercer [311]; the zones in the formation of the cortex were defined and the role of each was described. Marston [309] discussed the effect of cobalt- and copper-deficient diets on the formation of fibrous keratin in the wool fiber. Cobalt exerts its action primarily either on the lumen of the alimentary canal of the sheep or during its passage through the wall of the canal. Copper is more directly related to keratinization; a copper deficiency lowers markedly the quality of the fleece. Copper is therefore presumed to be primarily

responsible for catalyzing the oxidative closure of the thiol residues of the pre-keratin fibrous protein.

The decomposition of cystine and wool by ball milling and heat treatment was discussed by Stahl, McQue, and Siu [324]; the action of ultraviolet light as a photo-oxidative agent was discussed by Roberts [322]; and the affinity of water-soluble substances for wool was shown by Fischer, Seidenberg, and Weis [294] to be related to antibacterial activity—the greater the affinity for wool the greater the antibacterial activity for anion-active substances.

Bauer [281] made a study of weight losses occurring in the sulfuric carbonizing process and attributed these losses to the acid hydrolysis of the wool protein. The weight loss was considered to be a measure of the wool hydrolysis occurring in carbonization. In an analysis of the side-group constituents in wool, Middlebrook [317] estimated from colorimetric studies that the lysine content is 3.88% and the hydroxylysine, 0.18%. Tauber [326] described a new method for identifying tryptophan and related compounds; he used a color test for identifying the yellowish-green fluorescent compound to which tryptophan is converted as a result of perchloric acid treatment. It was pointed out that casein and other proteins that contain tryptophan give this color reaction, but that gelatin and silk do not. It is important to note that this color reaction is not obtained for any amino acid other than tryptophan.

3. Structure and Properties of Silk.—The literature relating to research on silk is not extensive in comparison with that on wool and synthetic protein fibers. Howitt [302] reviewed the structure of silk and discussed the amino acids obtained from both silk and wool by hydrolysis; the chemistry of silk and the structure of fibrous proteins were also considered. Zahn [330] made a study, using the electron microscope, of the fine structure of natural silk fibers in which the silk which had been treated with bacteria was first separated into individual fibrillae in a disc mill. These basic fibrillae are 250–500 Å. wide. Microtome sections of the untreated silk fiber, however, show very fine filaments of the order of 40–90 Å. wide. The ultraviolet spectrographic studies [295] indicated that for silk fibroin in 0.5% NaOH solution at pH 9.2 at room temperature tyrosine bands appear in the absorption spectrum. At pH 13 and 60°C, however, this absorption spectrum changes to that for glycyltyrosine anhydride, which is derived from the diketopiperazine in the fibroin. Fibroin from cocoons and silk glands of caterpillars [323] exhibit absorption bands characteristic of tyrosine in the region between 3,200 Å. and 4,500 Å. If the light vector is placed between 25° to 65° to the main chain axis, a sharp absorption band appears in the area 50–60 Å.; the origin of this band was not designated by the author. When radioactive glycine and alanine [332] were injected into the body cavity of a mature silkworm, the α-carboxyl groups of some amino acids associated with the silk fibers produced were radioactive. Further experiments indicated that the amino acids were incorporated into the protein of the living silkworm gland *in vitro*.

4. Synthetic Protein Fibers.—The α-β transformation in polypeptides was discussed by Bamford, Hanby, and Happey [278]. The entropy contribution to the load in stretched casein fibers was discussed briefly by Woods [327] and by Hoover, Kokes, and Peterson [300].

Arthur and Many [273] prepared fibers from cottonseed protein, for which the dry strength is of the order of 0.6 to 0.7 g. per den. and the wet strength of 0.2 to 0.3 g. per den. These fibers are yellow-to-orange in color and have a soft hand.

In work on regenerated keratin fibers, Mercer [312] studied the dissolved fraction of wool keratin, using saturated urea solutions containing reducing agents similar to those employed by Lundgren. The dissolved protein, salted out of solution, yields two kinds of precipitate; one precipitate may be drawn into fine fibers which have anisotropic physical properties and marked birefringence, and which yield the α type of x-ray pattern. Happey [298], in commenting on the α form described by Mercer for reconstituted wool, stated that the x-ray diffraction patterns of the casein fibers he studied did not show a typical α-keratin structure. He felt that the α form obtained by Mercer is probably due to the presence of protein which is not denatured in the solvents and which can be orientated in a matrix of soluble protein in a manner somewhat similar to that suggested for highly orientated casein fibers. Although certain polypeptide molecules produce x-ray diffraction patterns which are similar to that of α-keratin, the extent of this similarity has not been fully studied. Wormell and Happey indicated that it is probable that the α structure is not present in regenerated wool or in casein fibers [328].

Happey and Wormell [299], in a study of the structure and properties of keratin fibers regenerated from wool, gave a description of the methods for the dispersion of wool and its extrusion into continuous-

filament yarn with a β-keratin structure. The soluble protein is extracted with sodium sulfide, after which the protein is precipitated, redissolved, refined in cuprammonium solution, and finally reprecipitated. The yarn produced has a tenacity of about 1 g. per den. and an extension at break of 30% when tested at 65% relative humidity and 70°F. The physical properties appear to improve during processing. The authors suggested that this procedure for regeneration, which produces a β-keratin fiber from α-keratin, may be applied to waste products of the wool trade and to such other proteins as horn, hooves, nails, etc.

H. Structure and Properties of Synthetic Fibers

Numerous reviews describing new synthetic fibers have appeared in the literature [337, 357]. Azlon is the new casein fiber which was developed for bristles but which is now being used in air-cleaner felt with wool as a blending agent [340]. Celanese Corp. prepared a partially hydrolyzed acetate fiber called Celcos, which exhibits properties between those of viscose and acetate rayon [334, 335]. Dynel [338] is the Union Carbide and Carbon Company's version of the acrylonitrile-vinyl chloride copolymer similar to Vinyon N [336]. Polyacrylonitrile itself is being made into a textile fiber, which is to be distributed under the trade name "Orlon" [333, 349, 352].

There is considerable interest in the regenerated zein fibers, which are available under the name "Vicara" [346, 356]. This corn protein is dissolved in alkaline solution and reprecipitated in acidic salt solution, followed by a formaldehyde cure. The formaldehyde treatment improves the strength, elasticity, and dimensional stability of the regenerated fiber. Work to improve the solution and cure treatment is still in progress [348, 351].

Research on the development of new synthetic fibers has been fairly active in recent years. Attention was given to the synthesis of polypeptides from amino acids, with significant results. Brown, Coleman, and Farthing [342] reported the formation of a copolymer of anhydro-*N*-carboxy-*DL*-alanine and *L*-leucine in the form of films and fibers with molecular weights as high as 15,000. Bamford, Hanby, and Happey [339] studied polymers of *DL*-β-phenylalanine and copolymers of this compound with *L*-glutamic methyl ester. They concluded that the polypeptide chains thus formed exist in a coiled structure because their x-ray pattern corresponds to that of α-protein. Reid and Daul [353] described the formation of a copper-aluminum carboxymethylcellulose fiber similar to alginic acid fibers. Schildknecht, Zoss, and Grosser [355] investigated polyvinyl alkyl ethers, as well as the polymerization of vinyl methyl ketone, *N*-vinylpyrrolidone, and *N*-vinylcarbazole.

I. High-Polymer Studies

Certain basic studies on the structure and properties of high polymers, although not directly related to textiles, are of interest and bear on problems encountered in research on textile materials.

The dynamic properties of high polymers as a general class of solids have received rather wide attention. Astbury [359] developed a stress-strain relationship for plastics, using the electrical analogy of a dissipative circuit. Ballou and Smith [360] measured the elastic and dissipative properties of Terylene (polyethylene terephthalate) yarn between 3 cycles per sec. and 30,000 cycles per sec. and related their results to parameters of an equivalent Voigt model. In a lower frequency range—1,000 to 6,000 cycles per sec.—Hillier and Kolsky [374] examined the dynamic elasticity and damping factors of polythene, neoprene, and nylon filaments. Use was also made of the sound velocity and attenuation in rubbers [375] and in liquid polymers such as polyisobutylene and poly-α-methylstyrene [383] in studying their bulk and shear properties.

Lyons [381] reported on studies of the dynamic properties of high polymers; he compared such widely different materials as steel, rubber, and silk, and presented a theoretical treatment of the dependence on frequency of the modulus and energy loss per cycle. Dunell and Tobolsky [366] proposed a stress-relaxation mechanism by which the dynamic modulus and internal friction of a textile fiber or rubber may be predicted from its static stress-relaxation behavior. Kolsky [380] described a method for determining the stress-strain relationship for materials when stresses are applied for periods of time of the order of 20 μsec., and discussed delayed recovery in terms of mechanical relaxation and memory effects of the material. A theory of the viscoelastic properties of high polymers was developed by Rossmassler and Eyring [387]. The theory was applied to experimental results obtained on Nylon 66 monofils.

The relationship of molecular structure to the physical properties of high polymers was examined by several methods. Considerable attention was

given to polythene and related materials. Brown [364] conducted x-ray studies of the stretching and relaxation of polythene; the effect of pressure on the volume, thermodynamic properties, and crystallinity of polythene was examined by Parks and Richards [385]. Stein, Krimm, and Tobolsky [395] conducted a thermodynamic and optical analysis of the behavior of polythene, polyvinyl chloride, and lactoprene, basing their conclusions about structure on the behavior of the stress and birefringence with temperature. Spencer [393] considered the volume-temperature-time relationships for polystyrene and the viscous flow [394] of molten polystyrene. The effect of chain-branching in polystyrene on the osmotic pressure of polystyrene in solution was studied by Doty, Brownstein, and Schlener [365]. Morey [384] analyzed the viscous flow in polymers, basing his analysis on the dynamics of particle bonding and movement for non-Newtonian systems. He developed a theory of viscous flow, which is applied to dry polyvinyl acetate to explain the increase in activation energy of flow with increasing temperature. The intrinsic viscosity of a polyvinyl acetate solution was related to its adhesion to regenerated cellulose films and to aluminum foil [382]. Studies of the cross-linking process in gelatin gels [368] were conducted by measuring the specific rotation and rigidity for 5 degraded gelatin samples in the temperature range 0°C–40°C. Hill [373] pointed out that the melting points of paraffinic hydrocarbon polymers tend to approach asymptotically that of polythene. The influence of odd and even components on the melting points of the polyamides was traced to the changes in position of those groups contributing to the formation of hydrogen bonds. Elliott, Ambrose, and Temple [367] detected double orientation in hot rolled sheets of nylon and polyvinyl alcohol by using polarized infrared radiation with an angle of incidence of 54° to the plane of the sheet. Kolb and Izard [378] established the relationship between intrinsic viscosity and dilatometric properties of polyethylene terephthalate (Terylene), and discussed the effect of crystallinity and copolymerization on the temperature of the second-order transition. These authors [379] also used the dilatometric approach to measure the rate and amount of crystallization of polymers, particularly polyethylene terephthalate.

Theoretical studies of a general nature on mechanical properties were reported by Rivlin [386] on the problem of flexure in large elastic deformations of isotropic materials, and by Scott-Blair and Caffyn [392] on the application of the theory of quasi-properties to the treatment of anomalous stress-strain relationships.

In the field of polymerization and depolymerization (degradation), Whyte and Melville [396] conducted experiments on photosensitization of the polymerization of vinyl acetate and methacrylic acid. Studies on the thermal degradation of polyvinyl chloride [371], polystyrene [376, 377], and polyethylene [377] were reported.

The nature of the mechanism of plasticizer action in vinyl chloride systems was considered by Havens [372], and by Alfrey, Wiederhorn, Stein, and Tobolsky [358].

Additional papers in the general field of high polymers included studies on the formation and properties of three-dimensional polymers [361–363], polymer chain configurations [369], the thermodynamics of high polymer systems dealing with phase equilibria of solutions [390, 391], and the theory of crystalline states and fusion in polymers, copolymers, and their mixtures with diluents [370]. For cellulose nitrate the mechanical properties have been related to the shape of the D.P. distribution curve [388, 389].

J. Fiber, Yarn, and Fabric Inter-Relationships

Because fibers are twisted into yarns and strained in this twisted state, research on the torque-twist relationships in filaments and yarns is important. Morton and Permanyer [419] emphasized the need for uniform fibers in order to avoid, at the fiber thin places, torsional strains exceeding the yield strain of the fiber. When twist is put into a multiple-filament system, the strain is greatest on the filaments following the helical path about the yarn; any filaments in the core position have a shorter path to follow and, because of buckling, make a smaller contribution to the total torque in the yarn specimen than do the helical filaments. The ease with which the fibers fit into the helical form depends upon fiber flexibility [410], a property which is improved by wet-twisting [399], which gives stronger and more compact, but less pliable yarns. With staple fibers, fiber length and strength, as well as twist factors, are important in determining yarn properties [401, 404, 431].

Production of extremely lightweight yarns and fabrics is made possible by the use of fiber blends containing a soluble yarn [418]. One such yarn is that made from a metallic carboxymethylcellulose derivative (see Reid and Daul [353]).

Methods of assessing variations in yarn diameter [398] and of eliminating yarn faults were reviewed by Townsend [433] and by Pomfret [421]. Jackson [411] advocated the square of the diameter or the cross-sectional area of the yarn as a better measure of yarn variation than diameter alone.

A. S. T. M. adopted the Grex system [424] for yarn numbering.

Research in 1949 demonstrated that fabric-design problems lend themselves to mathematical treatment with increasing success [415, 420, 422]. Shorter treated the technique of formal textile design as an enumeration problem in mathematics [426, 427]. The problem of strain distribution in fabrics as a special field of textile mechanics was approached by means of a "trellis" model, which was shown to be applicable to textile fabrics despite their visibly discontinuous structure [405, 428, 429, 435].

The problems in designing and evaluating fabrics for various special uses were given considerable attention [408, 412]. Fabric requirements for arctic wear [409, 412], comfortable apparel [417], and cleanable clothing [407] were all examined. The influence of fabric structure and fiber abrasion on the felting of fabrics made from smooth fibers was also studied [402].

Methods were described for measuring the thermal transmission [438], pore-size distribution [403, 434], abrasion characteristics [406], and water-resistance properties [414] of fabrics. In the last case it was found that data obtained with fabric holders in the laboratory correlate quite well with the results of tests on garments worn by human subjects in the rainroom. Abrasion-testing problems continued to be of interest [423, 430]. One of these which received a great deal of study was the abrasion of webbing for military applications; nylon was found to have many desirable properties for such usage [437].

Interest in the infrared drying of textile products continued [432]. Wilhelm and Smith [436] studied the transmittance, reflectance, and absorptance of textile materials in the near-infrared wave-length region, and obtained results which are of value in problems concerning the comfort qualities of clothing, as well as in the use of infrared-drying techniques. The efficiency of infrared-drying plants was also considered [397].

Lamb, Albrecht, and Axilrod [413] surveyed the use of textile materials in plastic laminates. Their report deals with the flexural, tensile, and compressive properties of such materials between −70°F and 200°F. Other bonded fabrics and yarns were also reported [400, 425]. Such materials were found useful in the electrical industry as insulators [416].

K. Equipment and Techniques

The postwar advances in electronics and radiochemistry quite naturally continued to influence the development of equipment and techniques in the textile industry. Other advances in technology were brought about through the better understanding of the physical and chemical properties of textile materials. Because of the vast scope of the equipment and methods developed during 1949, no attempt has been made to present an analysis of all of the contributions.

1. Test Instruments and Methods.—The trend in tensile testing of textiles in recent years has been toward the examination of individual fibers and filaments. This has been the result of two factors: the realization that single-fiber testing is necessary both for fundamental research and for development in textiles; and the fact that advances in technology have made single-fiber testing possible. Development of numerous devices for single-fiber testing, incorporating various electronic strain gages, was reported in 1949. Dart and Peterson [479] described a constant-rate-of-extension fiber tester which employs an unbonded resistance gage. Reichardt, Schaevitz, and Dillon [453, 530] reported on an instrument which utilizes a ring dynamometer and a linear variable differential transformer which has extreme sensitivity and stability. A servo-controlled tensile tester which utilizes bonded strain gages in conjunction with stress beams and weight bars was developed by Instron Engineering Corp. [442, 449, 474]. This instrument, with a load range from 2 g. to 5,000 lb., may be employed for load-elongation measurements on single fibers, yarns, ropes, fabrics, and heavy test specimens.

The growing interest in dynamic studies of the elastic properties of textiles was shown in papers by Hillier [492], Schneider and Burton [535], Barach [460], Smith, Ferry, and Schremp [543], Breazeale and Whisnant [470], and Stanton [544].

The limitations of the normal elongation-to-break measurements obtained on conventional testing devices were illustrated by the improvement of standard testers [487], the comparison and assessment of normal tests [465, 525], new methods of testing [450, 486, 499, 519, 528, 549], and new apparatus for tensile testing [448, 547].

Measurement of the frictional and torsional elastic properties of fibers has been coming to the foreground in textile research. Recent work on the determination of the torsional modulus of wool was described by Lochner [504]. Kock [496] made a study of the break-twisting angle, and reported on its usefulness as an index of fiber brittleness. Makinson [506] discussed the work of Lindberg and Gralén on the frictional properties of wool fibers as determined by the fiber-twist method, and Moncrieff [514] reviewed the methods of measuring the directional friction effect of hair. Makinson [507] also published a note on the theory of the lepidometer.

Two new abrasion-testing machines were developed under the auspices of the National Bureau of Standards [534] and the Quartermaster Textile Materials Engineering Laboratory [546]. Results of tests performed on the latter showed good correlation with field-test results.

Roseveare and Waller reported on a dynamically balanced fatigue tester for rayon tire cords [531].

A ballistic method for the determination of interply adhesion was developed by Millard [510]; this method was also discussed by Davis [480]. Anderson described a tensometer for measuring the peeling force of multi-ply fabrics [439].

Tallant [550] discussed the use of probability paper for estimating the mean and standard deviation of normally distributed test data, while Chakrabarti [476] illustrated methods for estimating the standard error and coefficient of variance, with special reference to yarn strengths. Coghlan and Perry [477] considered the analysis and interpretation of end-breakage data in preparation and weaving. Ingham [494] reviewed the factors involved in planning a mill experiment, with particular reference to worsted processing.

2. Control Instruments and Methods.—The application of controls to the textile industry begins with the examination of the raw material and continues through the rigid operating conditions in the mill to the examination of the finished products in the laboratory. This field is therefore extremely diversified in scope. For example, control of the uniformity of materials is an almost continuous operation throughout every process in the manufacture of all finished textiles, whereas testing for water-repellency is specific for only one type of finished textile. For reasons of this kind, the material reviewed here on control apparatus and testing does not allow for homogeneity of context.

The control of uniformity in a textile mill begins with the examination of fiber length and diameter and with the determination of strength of individual fibers and bundles of fibers. Ramsthaler [529] discussed the fiber-strength distribution in mixtures of staple rayon. Fiber-length studies were reported by Bohringer [464]. Attention was paid to the method of determining fiber diameter with an air gage [440, 441], as well as to the statistical analysis of fiber-diameter measurements [522] and the evaluation of evenness tests [447].

Measurement and control of variations in the cross section and weight of slivers, rovings, and yarns were given much attention, as evidenced by the new instruments developed for measuring uniformity. Although both pneumatic [462, 463] and mechanical [454, 545] methods have been developed, the majority of the instruments are based on the dielectric principle [446, 455, 468] or on the use of photoelectric circuits [458].

The final control concerns the uniformity of the finished fabric; examination at this stage is exacting work which depends largely on human judgment. Eyestrain in the examination of finished fabric will be reduced through the use of a cloth-inspection machine [445].

The determination of the moisture content of textile materials is a difficult undertaking, principally because no clear-cut definition of moisture content with reference to bound and unbound water has been universally accepted. Furthermore, it is not known to what extent each of the various methods of determining moisture content removes the bound or unbound water. Each of the various methods, however, may be calibrated empirically to give the optimum results desired. The various types of moisture testers were described and evaluated by Moncrieff [515–518], Seaman, McComas, and Allen [538], Toner, Bowen, and Whitwell [551], Whitwell and Toner [557], Balls [459], and Hearle and Jones [491].

Moncrieff [512, 513] also discussed the theory and application of pH measurement to the textile industry.

A device for measuring and recording automatically the tension on a yarn as it is wound on a bobbin was developed in Canada [443]. Methods for testing sizes used for continuous-filament yarns of high filament strength were described by Bradbury [469]; Moncrieff [511] discussed the measurement of twist. The Fade-Ometer was critically examined with re-

spect to conditions of temperature and humidity in the vicinity of the test samples [520]. Recent advances in low-temperature pyrometry allow temperature measurements to be made on moving textile materials [467].

3. Identification and Examination.—Douglass and Crossmon [482] reported on the identification of fibers by the "dispersion staining" method. Preston [526] and Preston and Saha [527] discussed the identification of fibers by determining the temperature of contraction and by froth flotations. The tinting and fugitive identification of textile fibers was reviewed by Uhler [555].

Discussions of the use of the spectrophotometer as an aid to dyeing [542], the application of pH and spectrophotometric control in dyeing and textile operations [489], and spectrographic studies of treated textiles [451, 452] summarized the importance of the spectrographic examination of textiles.

Several new automatic recording infrared spectrometers [472, 556], and recent developments in the field of infrared spectrography [540, 548], including the role of the reflecting microscope in infrared spectrography [461, 539], were reported.

The increasing importance and use of radioisotopes in the examination of textiles was evidenced by the number of papers reporting their application [457, 466, 488, 493, 495, 497, 505, 558]. The chromatographic method of analysis was used extensively in cellulose chemistry [471, 473, 475, 478, 500, 503, 521, 533].

The use of the electron microscope [484, 524, 537, 554] and x-ray apparatus [456, 552, 553], including small-angle scattering techniques [498], in the examination of textile materials provided important information concerning the structure and morphology of textiles. Lindsley, Fischer, and Brant [502] showed that soft x-rays can be used to form shadowgrams of textile fibers, yarns, and fabrics on photographic plates. Single fibers of rayon, wool, and cotton are clearly resolved by 5 kv. x-radiation. Fibers impregnated with lead sulfide and other absorbing components can thus be distinguished in masses of untreated fibers; this technique provides a possible means for tracing individual fibers through processing operations.

Several discussions of the application of photography to the study of textiles appeared [501, 508, 541]; an interesting paper in this field dealt with a diagonal-sectioning technique for studying fabrics [523]. Other papers of interest covered the following subjects: an index of the degree of water-repellency [536], the evaluation of fire-resistance [532], a new color-matching unit [490], density measurements of fibers [509], the design of a transmission type of interferometer microscope [483], the determination of the alkali solubility of wool [559], and a method of measuring the surface tension of viscous liquids [485].

V. Development

This section of the review has been divided arbitrarily into "Manufacturing Methods" and "Finishing." The former includes papers on general developments in production and control techniques, and such special categories as carding; sliver and top; drafting, spinning, and twisting; sizing; weaving; and yarns and fabrics. The division on "Finishing" deals with wetting, detergents, and scouring; bleaching; dyeing and printing; shrink-resistance; crease-resistance; and fire-resistance.

A. Manufacturing Methods

1. General Processing and Control.—In the development of instrumentation, there was a marked trend toward the use of automatic control equipment. The measurement and control of relative humidity and moisture regain in yarn and cloth was improved by using automatically operated equipment [562]. Methods for improving the control of blending of fibers through accurate weighing [560, 561] and better control of tension and velocity of web material by means of a sensitive hydraulic-controlled variable speed unit [566] were described. Important factors in the selection of a temperature-measuring system for textile mills were reviewed by Bowden [569]. Some attention was given to the problem of the drying of textiles and to those factors which are important in the control of drying equipment [584, 585]. Use of artificial daylight in the textile industry was reported to offer advantages [574, 587]; these include easier fiber identification and better determination of wool damage and contaminations which are not normally visible with sunlight. The effect of electrical static in textile processing was considered by Lehmicke [576]; Lester [578] pointed out the role of photographic engineering in textile research. For high-speed machine sewing operations, Hume and Lee [575] described a method for the dry-cooling of needles which makes it possible to undertake heavy-duty sewing operations without overheating the needles. In engineering developments

relating to equipment using rolls or cylinders, a method was described by Prechter [580] for determining the inertia of a roller if the weight, maximum diameter, and core diameter are known.

Papers concerned with general processing and production dealt with spun-silk production [571], advances in the cleaning of cotton at the gin [572], flax processing and quality [573], and the effect of processing on the volume of several kinds of cotton, wool, and rayon staple as affected by pressure [579]. Some general observations on textile processing in the United States were presented by Smith [582]. Prentice [581] gave a brief account of the use of chemistry in the entire textile processing field, including wool scouring, piece and stock dyeing, carding, spinning, and finishing. Stoves [583] outlined three methods for setting wool under tension by treatment with hot water and steam; he also presented a theory of the setting process under the same conditions. For several operations in the processing of wool, Leonardsen [577] described the use of olein as a wool-oiling agent.

Bouvet [568] reviewed the factors which affect quality and production in rayon mills and described the effect of relative humidity and tension in the processing of rayon. The stretching of freshly extruded filaments of rayon was considered to increase greatly their tensile strength [563]. The processing of rayon cut staple [564] was discussed. The use of nylon staple on woolen and worsted systems was considered from the point of view of general processing conditions and the usefulness of nylon and wool in blends [565, 567]. Wignall [586], however, cautioned that in some cases nylon can be overused in blending with wool.

2. Carding.—Martindale [592] reviewed the evolution and early development of carding machines. Strang [593–595] developed an aerodynamic theory for the forces acting on fibers during carding; he analyzed the influence of air currents on carding, and of the air shearing forces on fiber separation, and discussed the control of air flow by the flats. The differential action of a card on the long and short fibers of a blend was considered by Hind [591]. Townend [597] described the recent research on the carding of wool at The University, Leeds, England. The role of the woolen carding condenser and its effect on slubbing variation were reviewed by Thorndike [596]. Dorman and Pringle [590] outlined the problems relating to the carding of flax, hemp, and jute; their influence on the design of the bast fiber card was shown. The suitability of these machines for the carding of rayon staple was discussed in this paper, and the modifications necessary for rayon-staple carding were suggested.

3. Sliver and Top.—Morton and Summers [603] described experiments, using a dyed-"tracer"-fiber technique, for determining the fiber arrangement in card sliver. The formation of sliver by carding and by other methods was reviewed by Hardacre [601]. He considered the carding of rayon staple on worsted, flax, and jute cards, and mentioned the development of machines for the conversion of continuous tow into sliver form in one operation, eliminating the processes of carding and combing. Bowden [599] described a new sliver tester. Methods of wool top analysis were described by Beevers [598], Haigh [600], and Wadley, Pohle, and Mueller [604]. Lang [602] considered the effect in top-making of the variability of fineness of Merino wool.

4. Drafting, Spinning, and Twisting.—Cox [609] developed a theory of drafting of wool slivers. The theory embodies a mathematical study of the dynamic mechanism of drafting, the purpose of which is to determine those factors which affect the irregularity of the yarn produced. A study of the possibilities of utilizing wool sliver from a Noble comb as a raw material for the drawing process was carried out [616]. The use of such a thin sliver allows the omission of the majority of the open drawing processes. The yarns produced in this way compared favorably with those produced from the same (64's) wool by the normally accepted top-finishing and drawing processes; the comparison was based on the long- and short-term irregularity of the yarn and on the yarn strength.

The repeatability and tolerance of laboratory spinning techniques [615] were studied by Stewart and Parsons. Pringle [613, 614] discussed the theory and practice of spinning. The spinning of nylon-wool blends [607] led to the interesting result that each 1% addition of nylon staple provided a 3% increase in strength in a woolen-spun yarn or a 3½–4% increase in a worsted yarn. It was also pointed out [605] that knitted nylon fabric waste can be successfully spun with wool. The spinning of rayon staple and the possibilities of fiber blending with rayon staple were also reviewed [606].

Dakin and Smart [610] described the principles of double twisting and reviewed the literature on the subject; Hargreaves [611] gave an historical review of the development of uptwisters for the twist-

ing of continuous-filament synthetic fibers. Bogdan [608] made a study of roving coil spacing on bobbins in which he derived an equation for the calculation of roving coils per inch from a given roving number for American carded cottons. He also discussed relative roving density, width of coils, and rates of change, and applied this general study to a specific manufacturing problem. Jones [612] reviewed briefly the various methods for drying warps at the tape frame and pointed out that cylinder drying appears to have many practical advantages.

5. Sizing.—Seydel [624, 625] made an extensive review of the problems relating to cotton warp sizing. He presented an extensive bibliography [625], covering the literature of the past twenty years, on cotton warp sizing and related subjects. The study of the effect of sizing on warp breakage was discussed by Bradbury [618]. He reported some results obtained in the sizing and weaving trials at The Shirley Institute over a number of years; these results were classified, and a working hypothesis of the causes of warp breaks was suggested, which appears to be helpful in explaining some of the effects observed. The advantages of the cotton slasher with respect to spun yarns were discussed [622] and mention was made of a suitable size product which can be adapted for general use with fiber combinations which are difficult to size. The use of polymethacrylic acid for sizing nylon filament yarn was discussed [617], and formulas, mixing instructions, and effect of the size on yarns during weaving were outlined. Urea as a solvent aid for cellulose textile sizes [627] was evaluated; urea was not found to be an economical aid for alkali-soluble ethylene oxide ethers of cellulose, although it was found to reduce the solution viscosity.

Bradbury and Hacking [619] worked out an experimental technique for making mill studies of the effect of sizing treatments on warp breaks in weaving without serious disruption of mill routine. An electronic size-cooking control system was described by LaVoie [621]; this system offers the advantages of producing an increased uniformity of size solution from batch to batch, effecting better utilization of the time of skilled operators, increasing batch production, and eliminating "boil over" when the cooking temperature is approached. Rainard and Zanolli [623] described a direct steam-injection continuous starch-cooker and the characteristics of pastes produced with this type of equipment.

The use of homogenizers in the preparation of sizing solutions for warp yarns is a new application in the textile industry that appears to offer many advantages. It is claimed that homogenization eliminates the need of boiling, makes a size which does not separate out on standing, and produces a much more uniform viscosity, leading to more even application of the solution to the yarn and thus increasing the weaving efficiency. Kennedy [620] described several types of homogenizers and mentioned the experiences of a number of mills in their application.

6. Weaving.—The causes and effects of warp breakages in weaving received considerable attention during 1949. Baines and Steiger [628] discussed the effect of warp breakage and loom efficiency in the weaving of plain cotton fabrics on Lancashire looms. Brown [633] considered the loss in productive capacity due to loom stoppages caused both by warp and weft breakages. In the weaving of continuous-filament and long-staple spun yarns, Castle and Dawson [634] discussed the factors affecting weaving efficiency for nett silk, spun silk, and nylon warps. The number of warp breakages in woolen and worsted weaving was attributed by Snowden [637] to the lack of strength and extensibility of the warp yarn itself, loss in strength and extensibility of the warp yarn as it passes from the warp beam into the fabric, and the tensions and stretch imposed on the warp yarn during weaving. Vincent [638] reviewed the results of research conducted during the past 20 years at The Shirley Institute on the effect of varying certain loom settings on warp breakage. The relationship between loom settings and fabric structure was discussed by Barker [629]. A rapid stroboscopic study of production looms [635] was described in which looms were checked rapidly, with a minimum of interference to mill operation, while running under normal illumination.

In discussing the requirements for the preparation of warp yarns for automatic weaving, Sheldon [636] named efficient methods of yarn preparation, warp breakage analysis, and automatic control of warp-beam tension as the major factors in efficient loom production. In the beaming of warp yarns [630] strain-gage tests were used in order to determine the stress applied to the warp beam. It was pointed out that nylon exerts the greatest crushing stress on the warp beam, with stresses as high as 7,000 lb. per sq. in. In the processing of continuous-

filament viscose rayon, the effects of tension in the processing and the effects of warping and warp sizing on quality were reviewed by Bouvet [632]. Blackburn and Sondhelm [631] compared the Barber-Colman super-speed warping equipment with the Lancashire beam-warping equipment in order to study the number of stoppages per unit length of yarn warped on the conventional slow-speed warping frames with the corresponding figure for the super-speed beamer and to find the causes of the stoppages. The results indicated a large reduction in the number of warp stoppages obtained with the Barber-Colman equipment, and preliminary results suggested that the Barber-Colman-prepared yarns have a considerably lower end-breakage rate during weaving than do the Lancashire-prepared yarns. Another advantage of the Barber-Colman beamer is the magazine system of creeling.

7. Yarns and Fabrics.—The influence of fiber properties on yarn strength was treated in a series of articles by Dakin [647, 648]. Viscose-filament yarn production [649] was reviewed from an economic point of view, whereas the development of and uses for nylon yarns, both staple and continuous-filament, were outlined [645]. Studies on the processing of woolen and worsted yarns included considerations of humidity control [640], the production of low- and medium-count yarns on the American system [639], and the processing of worsted-type yarns using uncombed wools [656]. The influence of mixed fibers on yarn and fabric design [642] and the effect of tension in the early and later stages of processing of yarns and fabrics were considered from the viewpoint of the quality of the finished products [643].

Tripp [654] reviewed the trends in fabric processing. The National Cotton Council of America made a survey of the use of cotton in nonwoven fabrics [644]. The effects of fibers, bonding agents, and finishing treatments on bonded-fiber fabrics were considered in a review by Bromley and Elliot [646] on the manufacture of bonded fabrics from card webs. The effect of filament denier of the monofils and the characteristics of nylon fabrics were reported by Vessie [655]. Several different methods of heat-setting nylon fabric, based on a wide variety of end-use requirements, were considered [641]. In rayon fabrics the necessity for insuring satisfactory performance characteristics was stressed by Tattersfield [653]; he discussed some of the difficulties associated with the laboratory estimation of the behavior of these fabrics in use. Standards of performance with reference to resistance to light and washing were tabulated for 47 items of wearing apparel and household textiles.

Other papers relating to the field of fabric developments were concerned with problems in textile fabric defects [650], precision control of moisture content in tentered fabrics [651], and cloth construction and fabric analysis [652], in which plain, twill, and satin weaves were considered.

B. Finishing

The general papers in this broad field concern chemical problems arising in textile finishing [659, 674], the application of special chemicals as finishing agents—such as thermoplastic and thermosetting resins [669] and their curing agents [657]—, the application of rubber to wool [663], the use of waxes in finishing, dyeing, and printing [666], and the use of special chemical compounds [671, 675, 676]. Turner [670] reviewed the developments in the design of machinery used for the dyeing and finishing of cotton and rayons.

The function of a laboratory in the evaluation of textile finishes was considered by Cook [661]. The author discussed several methods for evaluating textile finishes, with particular reference to softeners; he also considered the effect of these finishes on dyes, on the tensile strength of textiles, and on human skin. Schaeffer [667] made a study of the use of the rH value in processing control. The rH value was originally proposed by Clark as an indicator for oxidation and reduction processes. Schaeffer described practical measurements made in textile finishing and indicated that these measurements may acquire great importance for the control of operations in vat dyeing if it is possible to bridge the existing gaps in the series of oxidation-reduction indicators used. The author presented tables of practical measurements for use in the field of vat dyeing. Cameron and Morton [660] presented evidence of the existence of cross-bonds in 9 viscose rayons which had been subjected to a urea-formaldehyde crease-resistance treatment. The change in fiber properties as a result of urea-formaldehyde treatment is due mainly to the cross-bonding of adjacent molecules, and the authors estimated that in the technically important modifications of viscose rayon there is one cross-bond for each 40–100 glucose residues of the fiber cellulose. It is interesting

to note that the authors reported that the cross-bonds differ widely in stability, and they pointed out the technical importance of this observation. Noble [665], in a study of the effect of temperature on finishing compounds (especially softeners), found that the cationic substantive softeners do not have as high a resistance to heat as the anionic types studied do. This conclusion was based on three types of tests: (1) accelerated ageing or drying, (2) rancidity, and (3) scorching. In recent experiments on cloth finishing, Whewell, Charlesworth, and Kitchin [673] noted that the setting processes for textile materials are influenced by pH. They commented on the scouring, felting, and production of unshrinkable wool fabrics and on the shrinkage of cotton-warp fabrics with various types of natural and synthetic fillings.

Additional papers in the general field of finishing include those relating to woolen and worsted fabrics [668, 672] and to fabrics made from synthetic fibers [662], including polyethylene [658] and nylon [664].

1. Wetting, Detergents, and Scouring.—Stadler [706] discussed the role of wetting in textile finishing and the methods employed to determine the efficiency of wetting. Edelstein and Draves [682] reported on a re-examination of present wetting tests and concluded that the choice of the wetting tests, either for control or for the comparison of wetting agents of the same chemical type, is essentially a matter of personal preference or convenience. Jensen [688] reported on the textile wetting agents in which the Danish textile industry is interested; in discussing practical measurements, the author pointed out that the determination of wetting power by surface-tension measurements is a valuable supplement to other practical tests in current use. The wetting efficiencies of a wide variety of chemical compounds for woolen cloth [689] and cotton fabrics [702] were discussed. Paice [699] described various types of wetting and detergent agents and reviewed their practical application in the textile and in other industries.

The application of soaps and synthetic detergents was analyzed by Schwartz [703] from a physicochemical point of view. The growing use of synthetic detergents in the textile industry, both for natural and snythetic fibers, received considerable attention [691, 698, 704, 705, 713]. Additional papers in this field include those dealing with the elements and theoretical considerations of detergency [685, 690], the use of laboratory techniques in the evaluation of detergents being used in the textile industry [693, 694], the adsorption of detergents by wool [677], and the use of sodium silicates in the textile industry [695, 696].

Utermohlen and Ryan [712] reviewed the various methods of measuring the effects of washing procedures on soiled cotton cloth; these authors and coworkers [711] examined the influence of oily soil upon the removal of pigment soil. Powney [701] studied the mechanics of the removal with detergent of oil adhering to fibers of wool, cotton, and kapok. The desoiling properties of carboxymethylcellulose were also reported [683].

Tripp [709, 710] discussed the use of auxiliaries for scouring before bleaching, together with various systems for continuous scouring and bleaching. For cellulose fibers, Hessler [687] tested dew-retted hemp for breaking strength and degree of polymerization after treatments with various degumming agents, including inorganic substances and alcohols. Parsons [700] studied the effect of alkaline scouring on the physical properties of dry-spun flax yarn and fabric. In the scouring of wool, Leonard and Beck [692] presented data on laboratory studies concerned with various factors affecting the efficiency of the scouring operation. A new instrument for reading directly the concentration of soda-ash solutions in wool scouring baths was described. The temperature of the scouring liquors appears to affect the behavior of the wool during its conversion into worsted yarn [708]. For Australian 64's, the wool scoured at lower temperatures suffered considerably less breakage in carding and gave a better test in combing and a greater mean fiber length in the top. The results on yarn strength were conflicting, so that it was not possible to relate the temperature in scouring to yarn properties on the basis of these tests. Heddergott [686] described three types of wool driers and experiments conducted on them, using the same wool under identical conditions.

2. Bleaching.—The field of textile bleaching was outlined in the textbook by Marsh [723]. Individual papers on progress in textile bleaching were written by Fargher [716], Hall [717, 718], and Klein [720]. The production, properties, and textile applications of the hydrosulfites [715] and the effect of various surface-active agents on the penetrating power and stability of calcium hypochlorite and bleaching-powder solutions [724] were the subjects of two

papers of practical interest. Methods for improving the continuous bleaching process were reported by Bowden [714] and Seyb and Foster [725]. In a study of the bleaching of blue cotton fibers, the apparent cause and location of the color in blue cotton was investigated. The factor which prevents the bleaching of blue cotton appears to be the lack of access to the blue color in the lumen by the bleaching solution. The diffusion of the blue color out of the fiber proceeded slowly when the fiber was placed in a mildly alkaline bath at elevated temperatures.

Photochemical methods for bleaching were outlined by Landolt [721], who described two new fluorescent bleaching agents—one which is suitable for bleaching cotton, and the other for bleaching wool, nylon, natural silk, and acetate rayon; by Livingston [722], on the irreversible and reversible photobleaching of dyes; and by Trevor [727], on the general subject of optical bleaching.

3. Dyeing and Printing.—In the practical aspects of dyeing, an interesting use of radioactivity was recently disclosed by the Vat-Craft Corporation—a uranium acetate compound is used in conjunction with a sensitizing agent, also containing uranium, in the dyeing of textile materials. The dyeing of cellulose fibers received considerable attention, particularly of cellulose fibers in blends with other fiber types. Papers dealt with the problems of dyeing the following blends: viscose and Bemberg [753], mixed acetate and viscose fabrics with vat colors [730], spun-dyed "Fibro" with cotton and wool [773], and nylon and wool [744, 758]. Asbury [741] studied the dyeing of hosiery with vat and naphthol colors and their solutions; problems in the dyeing of viscose rayon were reported by Armfield [740], Boulton [745], and Cluley [749].

In the dyeing of wool, Casty and Krähenbühl [747] discussed some new principles in connection with continuous dyeing; Millson [766] considered the effect of metals on tippy dyeing. The damaged tips of wool fibers have a preferential affinity for heavy-metal ions, and the presence of most of these in chrome dyes will produce colors different from those obtained with chromium itself. Concentrations of less than one part per million of these heavy ions in the dye bath may cause appreciable color changes. The application of carbolan dyestuffs [750] to wool yarn produces a leveling action which has not been possible with acid dyestuffs. Hug [757] described methods for applying vat dyes to wool. Jordinson [759] compared the mordant dyeing of silk with that of wool textiles; acid, basic, and direct vat colors were applied to silk, and the dyed materials were tested for fastness to light, washing, perspiration, dry cleaning, peroxide bleaching, cross dyeing, moist pressing, steaming, and discharge [729]. Luttringhaus [761, 762] considered the application of leuco esters of vat dyes to wool and the dyeing of helidons on wool. The prevention of wool dyeing faults and the special attention required in the dyeing of worsteds for hard wear were discussed by Marsden [764, 765]. Stevens, Whewell, and Yegenaga [777] studied the raising of dyed woolen fabrics in comparison with that of the same materials undyed. Breare [746] attempted to determine the effect of dyes on the photochemical oxidation of wool. The triphenylmethane group appears to offer the least protection against photochemical oxidation of wool.

In the dyeing of synthetic fibers, studies have been conducted on the application of chrome dyes to nylon [756], the dyeing of nylon from neutral dye baths containing magnesium sulfate [734], and the dyeing of broad-woven nylon fabrics [779]. The dyeing of Perlon with vat dyes was considered by Müller [768]. The Thermosol process for dyeing and printing Fiber V, Orlon, and nylon [739], and the surface dyeing of plastics [752] are of interest in the dyeing of synthetic materials.

Problems connected with the light-fastness of dyes and dyed materials were considered by Morton [767], Nuttall [769], Parks [771], and Smith [776]. A series of papers by Wright [785, 786, 787, 788, 789, 790] treated the problem of color adaptation and color contrast and gave a general account of the methods of investigation, the effect of the illuminant, and the problems encountered with dye mixtures.

In the field of techniques and instrumentation, a German method for pressure-dyeing applied to rayon in cake form [733] attracted interest, although it is apparent that more experimental work remains to be done. Davidson and Godlove [751] described the application of the automatic tristimulus integrator to textile mill practice. The Geigy chrome steam process [732] is of interest in the dyeing of nylon.

The screen-printing of textile materials and the techniques associated with these methods received attention by Beton [743] and by Wall [780–784]. Additional papers on printing included those dealing with general problems encountered in industry,

the choice of materials, and various processing techniques [735, 737, 748, 754, 770, 772, 774, 775].

4. Shrink-Resistance.—The majority of papers in this field were concerned with studies on shrink-resistant treatments for wool; interest was also shown in the control of shrinkage in cellulosic materials. Stoves [810] described the use of surface deposits, synthetic resins, and chlorination for shrinkproofing wool; he also reviewed the theories of the chemical action of chlorine and the properties of chlorinated wool. Chlorination processes for shrink-resistance were also briefly reviewed by Epelberg [794]. Studies of the shrinkage control of wool, based on fiber characteristics, were reviewed by Teplitz [811]; he discussed the wash-fast requirements and shrinkage control of fabrics made from various fibers. He pointed out that there are over 60 methods for shrink-controlling wool. Maresh and Royer [802] undertook a microscopical examination of methylated methylol melamine resin-treated wool fibers, and showed that the resin is located essentially in the scale region of the fiber. The mechanism by which the modification of this portion of the fiber produces shrinkage control is attributed to the change in the directional coefficient caused by the deposition of the resin at the scale interstices and to the change in the elastic properties of the fiber due to the formation of a semi-rigid sheath around the elastic cortex. This same resin treatment [801] also produces a marked increase in the torsional rigidity. Successive extensions of a treated fiber cause a gradual decrease in the rigidity, which approaches that of the untreated fiber. It was concluded from this study that resin treatment affects only the surface portion of the wool fibers and that this modified surface structure must be chiefly responsible for the shrink-resistance of the treated wool.

The factors contributing to cloth shrinkage were reviewed [791]. Methods for reducing this shrinkage and eliminating felting were discussed by Gould [797]. The control of shrinkage by resin application was reviewed by Moncrieff [805], who discussed the Resloom process, the use of silicon tetrachloride, and the mechanism of resin anti-shrink finishes. The Lanaset process was discussed by Moncrieff [804]; a new process for applying Lanaset resin to wool at low temperatures [792] appeared to be applicable to a wider range of fabrics, with more exact control and less resin used. Methods for wool-shrinkage control using resin treatments included processes for applying films of cellulose acetate and the use of anhydrocarboxyglycine [803]. Processes which employ urea-formaldehyde resins were reviewed by Shapiro [808]. The function of methylated melamine-formaldehyde resins in the control of shrinkage of woolens was studied by Stock and Salley [809], who compared resin-treated and untreated fabrics and found that the difference in minimum strength of the yarns appears to be related to the logarithm of the ratio of the shrinkages. Lipson [799] considered the problem of applying synthetic resins to wool and developed a new method for forming vinyl polymers in wool [800].

The Quartermaster Corps [812] reported the development of an accelerated test method for predicting the amount of shrinkage that a fabric undergoes in laundering. The characteristics of this test indicate that it may be applicable to practically all of the types of shrink-resistant treatments now commercially available. The test appears to be reproducible within very narrow tolerance limits, and a very good correlation was found to exist between this accelerated test and successive Army mobile launderings for the four different types of resin-treated fabrics studied. The dimensional changes of knit goods in laundering were also studied [796] and were found to be related to the construction, the number of courses per inch, and the fiber type. Knitted fabrics usually shrink more in length than woven fabrics of similar fiber content do, and it is reported that the greatest change usually takes place in the first laundering. Fletcher [795] found that for cotton jersey the dimensional stability fluctuates according to the number of courses and method of laundering. The lengthwise shrinkage increases inversely with the number of courses, whereas the width shrinkage increases in direct proportion to the number of courses. If these fabrics are tumbled during laundering and drying, the length shrinkage is greater than when they are not.

The shrinkage of cotton yarn in zinc chloride was studied by Kasbekar [798]; the factors causing shrinkage in rayon fabrics and nylon were reviewed by Pfeffer [807]. The relation of swelling to shrinkage was discussed by Munch [806].

5. Crease-Resistance.—Buck and McCord [814] conducted a comprehensive study of the treatment of textiles for crease-resistance. The influence of fiber structure and fabric construction were considered in relation to the problem of creasing and wrinkling.

Crease-resistant finishes were reviewed, together with problems encountered in fabric and resin preparation, application of resins, drying of the fabric, resin curing, and washing of the fabric. Test methods for evaluating crease-resistance were also outlined; sketches of several types of equipment used in crease-resistance studies were given. An extensive bibliography is included in this review. Nickerson [819] also reported on anti-crease treatments for cotton. Nuessle [820] discussed the practical application of synthetic resins to cellulosic textiles. Gagliardi and Nuessle [816] considered the modification of fiber and fabric properties by wrinkleproofing and stabilizing agents. Moncrieff [818] considered crease-resistance from the viewpoints of the mechanism of creasing, the reduction of the tendency to crease, and cross-linkage.

Crease-resistant finishes containing urea-formaldehyde resins were under investigation. Shapiro [821] discussed the effects of fiber, yarn, and fabric structure on crease-resistance. Ferrari [815] studied the strength of fabrics which had been treated with urea-formaldehyde resins, and pointed out that the loss of tensile strength of cotton in this creaseproofing process is due partly to mechanical effects, such as the loss of elasticity by fiber incrustation, and partly to chemical effects, such as the action of the acid catalyst, particularly at elevated temperatures. Gruntfest and Gagliardi [817] studied viscose-filament yarns and wool fabrics which had been treated with three different urea-formaldehyde resins, with formaldehyde, and with glyoxal, and compared the physical properties of the treated fabrics and yarns with those of the untreated materials.

6. Flameproofing.—Buck [822] reported on an interlaboratory investigation on the flammability of textiles. This investigation was conducted in order to determine fabric types which are considered hazardous according to the method using the A.A.T.C.C. inclined tester and in order to determine the degree of correlation that could be obtained when a number of laboratories tested the same fabric on the inclined tester. Tests were also conducted on the horizontal tester. The results of this analysis indicated that laboratories differ significantly in burning times for different fabrics, that there appears to be no consistent pattern of differences between laboratories, and that the inclined and the horizontal testers do not show a correlation sufficiently high so that they may be used interchangeably. Weissberg and Kline [826] conducted simulated power-plant fire tests of doped fabrics which had been coated with fire-retardant coatings. These tests were conducted under wind-tunnel conditions and demonstrated that it is possible to increase the critical time interval between the instant of first contact of the fire with the fabric and the instant of fabric destruction. This interval is of the order of 2 sec. with cellulose nitrate dope, and 6 sec. with cellulose acetate butyrate dope; the interval may be increased to 12 sec. if a fire-retardant coating is applied over the cellulose acetate butyrate dope. A laboratory method was described for the quick evaluation of the relative performance of fire-retardant coatings. This test should facilitate the further development of coatings of this type.

Reid and Mazzeno [824] investigated various methods of phosphorylating cellulosic materials. Cloth phosphorylated with phosphorus oxychloride and pyridine is substantially flameproof and glow-proof but suffers such a great loss in tensile strength that the cloth is impractical for garments. This degradation may be avoided in the preparation of samples by using the urea-phosphoric acid method of phosphorylation. The authors presented data giving the effect of time, temperature, and molar ratio on the phosphorylation of cellulose, hydroxyethylcellulose, ethylcellulose, and cellulose acetate. These authors [825] also studied the structure of phosphates of cellulose which had been prepared by the urea-phosphate method of preparing flameproofed cloth and by the pyridine-phosphorus oxychloride method. By electrometric titration the combined phosphorus in cellulose phosphate (using the urea-phosphate method) is probably entirely in the form of a monosubstituted phosphate ester. The pyridine-phosphorus oxychloride method produces a similar monosubstituted structure, although in this case approximately 23% of the phosphorus can be accounted for as disubstituted phosphate ester. The chemistry of cellulose-fabric treatments with urea and phosphoric acid, followed by heating, was examined by Davis and Findlay [823].

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IV. Research

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Book Reviews

A Chemistry of Plastics and High Polymers. Patrick D. Ritchie. New York, Interscience Publishers, Inc., 1949. 271 pages. Price, $4.50.

(Reviewed by G. H. Fremon, Union Carbide and Carbon Corporation, South Charleston, West Virginia)

In addition to filling a need for "a more compact and moderately priced single volume dealing mainly with the organic chemistry of high polymers as a whole," the author provides in his preface a statement of aims and an appraisal of results that would be hard to improve upon. Those already familiar with the present book will recognize many of Dr. Ritchie's own statements in the comments that follow.

The book is intended primarily for organic chemists, and it is assumed that the reader's knowledge is of approximately graduate-student level. Condensation and polymerization reactions are covered in a general way in the first 3 chapters, and in the following 11 chapters each major class of commercially important high polymers is discussed in reasonable detail. The author does not pretend that his coverage of this vast field is complete ("it is not by accident that it is designated *A Chemistry* rather than *The Chemistry*"), and he follows his expressed intention to keep the synthetic and structural aspects of the subject to the fore, and to content himself with qualitative, largely non-mathematical treatment of the physics and physical chemistry involved. His handling of these latter aspects is adequate for his purposes. Such concepts as molecular configuration and orientation, close-packing, crystallinity, and hydrogen-bonding are introduced and in the chapter on the cellulose family these figure prominently in the discussion, and the next-to-last chapter is devoted to the relation between molecular structure and physical properties of high polymers.

Dr. Ritchie explains that his present work is based upon several courses of post-graduate lectures—he is Head of the Department of Chemistry, Birmingham (England) Central Technical College—and the book reflects its origins. It is obviously written by one thoroughly familiar not only with his subject but also with his audience, and his style of presentation is lucid, easy-flowing, and occasionally entertaining. In this latter connection this reviewer should perhaps confess that even if the book struck him as mediocre rather than first-rate he could not repress a cheer for an author who—to cite two examples—introduces his chapter on addition polymerization with a question by the White Queen ("What's one and one and one and one and one and one and one and one and one?") and disposes of the term "a plastics" as "an etymological monstrosity with no single redeeming feature."

Cotton Counts Its Customers. Memphis, Tenn., National Cotton Council of America, December, 1949. 30 pages. Free.

This report, compiled by the Market Research Department of the National Cotton Council, deals with the consumption of cotton and other materials by major end-use markets for the year 1947. It contains more information about the distribution of cotton and competing materials by market outlets than has previously been available from any one source.

The first part of the report lists the number of bales of cotton required to produce the quantity of a given article which was manufactured in its final form during the calendar year 1947. Cotton consumption is divided into 53 major classifications under "Apparel Uses," 20 items under "Household Uses," and 32 under "Industrial Uses"; there are subdivisions of many of the classifications.

An appendix tabulates the uses in terms of the total number of units manufactured, number of pounds, and the non-cotton content, with a conversion of the non-cotton content into raw cotton equivalent.

The general arrangement affords a quick bird's-eye-view for the reader having only a general interest, and the booklet is recommended for study by those persons engaged in market research and sales promotion.

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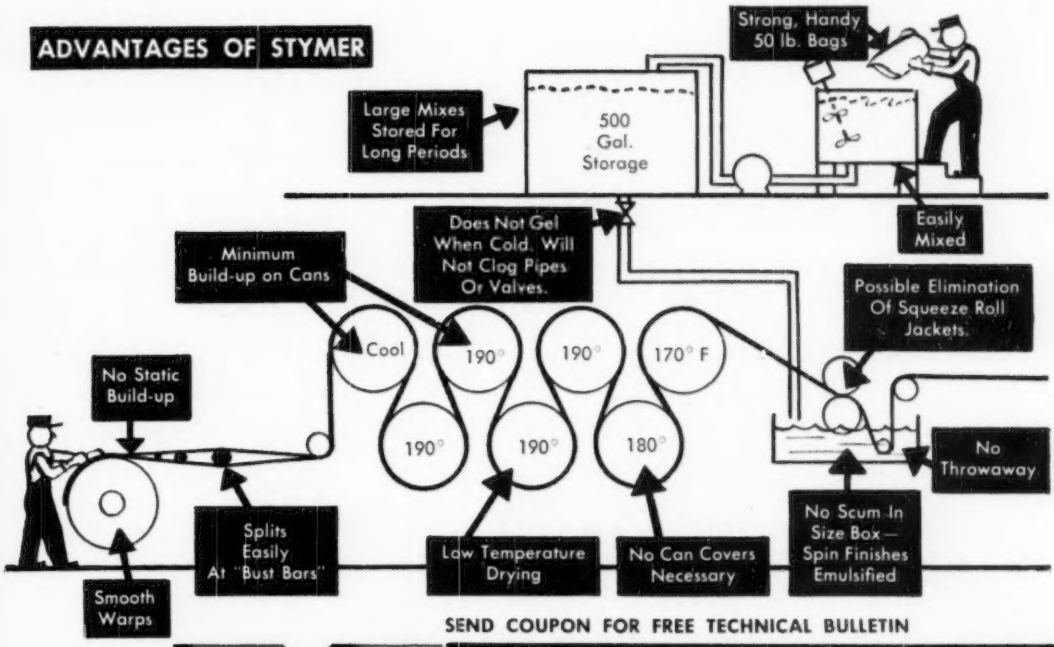

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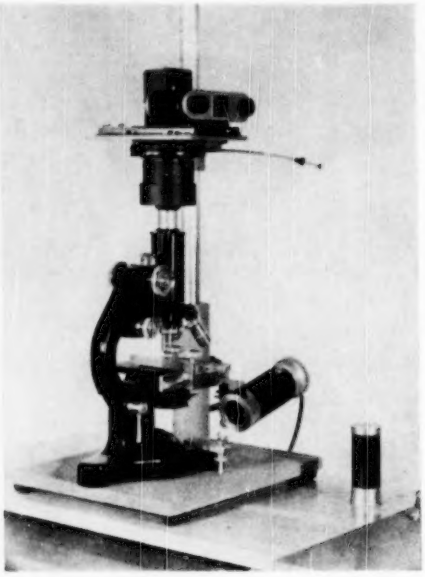

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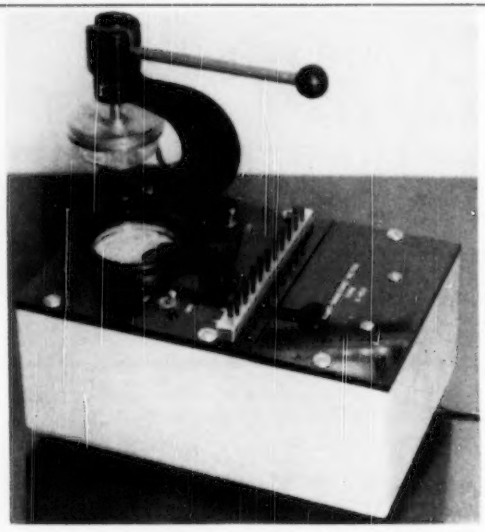

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